2339

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Date of request 10/10/95 Expected receipt of document ASAP

Document number CM-73 Date of document May 30, 1945

Title and author (if document is unnumbered)

LINK RECORDER OPERATIONS MANUAL — COPY ENTIRE DOCUMENT INCLUDING DRAWINGS + FIGURES. (PLEASE PLACE THIS REQUEST AT THE HEAD OF THE QUEUE). THANKS —

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UNCLASSIFIED

June 7, 1945

On-73

To: All Line Recorder Supervisors & Operators:

Please make the following corrections in your copy of the Operations Manual:

Page 115 - 100% Air Checks

Change steps #1 & #2 of the procedure to read.

1 - Open valves 92 and 91.

2 - Close valves 62 or 63, 99, 98, 96 and 83 or 84 in the order given.

Page 117 - 95 - 5 Check

Change steps 4 & 9 to read:

4 - Close valves 62 or 63, 99, 98, 92 and 91.

9 - When the check has been completed, close valves 80 82 or 78, 81 and the Calibration leak. Open 92 and 91 and pump out until the pirani is zero.

R. J. Thomas

R. J. Thomas

UNCLASSIFIED

June 7, 1945

To: All Line Recorder Supervisors & Operators:

For the past month, the Line Recorder Department has endeavored to maintain the cold traps in the stations as Dr. Nier has requested.

The requests are as follows:

1. No ice on the chemical trap finger or collar.
2. No ice covering the Manifold L-28 trap.
3. Keep covers on the Tube rack L-28 and slush traps at all times.

Adequate instructions as to the procedures has been covered previously, and as for means to scrape the ice off of the cold traps a 10¢ paring knife or any suitable sharp object will do the trick.

The above mentioned points are extremely vital for proper operation of the Line Recorders and any person willfully neglecting them will be required to account to me in person.

H. A. Nowak

H. A. Nowak

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May 31, 1945

To: All Line Recorder Operators and Supervisors

This is your copy of the Operations Manual, so write your name and badge number on the Title Page.

The manual has been written to standardize procedures through out the Department and only with 100% cooperation can this be attained. Follow the procedures given in this manual, and if at any time they become obsolete or are proven incorrect, revisions will be made. Periodically, additional information and the above mentioned revisions will be distributed for insertion in your manual. KEEP YOUR COPY UP-TO-DATE AND IN GOOD CONDITION.

Security regulations will not allow you to leave this manual lying around in the station or to take it out of the plant. Please act accordingly - we suggest that the manual be kept in your locker or in the station cabinet.

If any questions arise, explanations of the material presented herewith will be given by your supervisor or the Intra-Department Coordinator.

Because of the urgency in getting this manual finished, certain items such as page numbers, values of constants, telephone numbers, etc. have not been filled in completely. Included below is a list of additions which you should make as soon as possible.

Page	Add
7 ✓	~~Page 121 (cross out~~ the phrase "Not ~~indicated on the~~ Diagram")
8 ✓	~~Pages 109 & 117~~
18 ✓	~~" 136~~
20 ✓	~~" 140~~
21 ✓	~~" 132~~
~~101~~	~~" 102-108~~
~~103~~	~~" 106~~
~~117~~	~~" " 114~~
~~121~~	~~Number 121a & 122~~
~~124~~	~~Page 128~~
~~126~~	~~" 304~~
~~127~~	~~" 303~~
~~130~~	~~" 9~~
~~131~~	~~" 153, 147, 148~~
~~143~~	Could any of your L.R. Units operate correctly, under any conditions, with the Pirani reading as shown? Refer to Page 102.
~~150~~	Fill in these Phone numbers Office — Bell Phone — PAX 301-3 — 8-9286 — 45 303-5 — 8-9411 — —
~~152~~	~~Page 142~~
~~153~~	~~" 22~~
~~155~~	~~" 215~~
	2 " 151 ?
~~206~~	~~" 211a~~
~~211a~~	~~Number page~~
~~212~~	~~Page 215~~

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Respectfully yours

M. Burg
R. J. Thomas

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LINE RECORDER DEPARTMENT

OPERATIONS MANUAL

Compiled and Edited

by

R. J. Thomas

and

M. Burg

Contributions were made by various members of the Line Recorder Department Supervisory Personnel.

May 30, 1945

meg

[redacted]

Copy Number 128

Owner ~~Stella Ball~~ Norton Gerter

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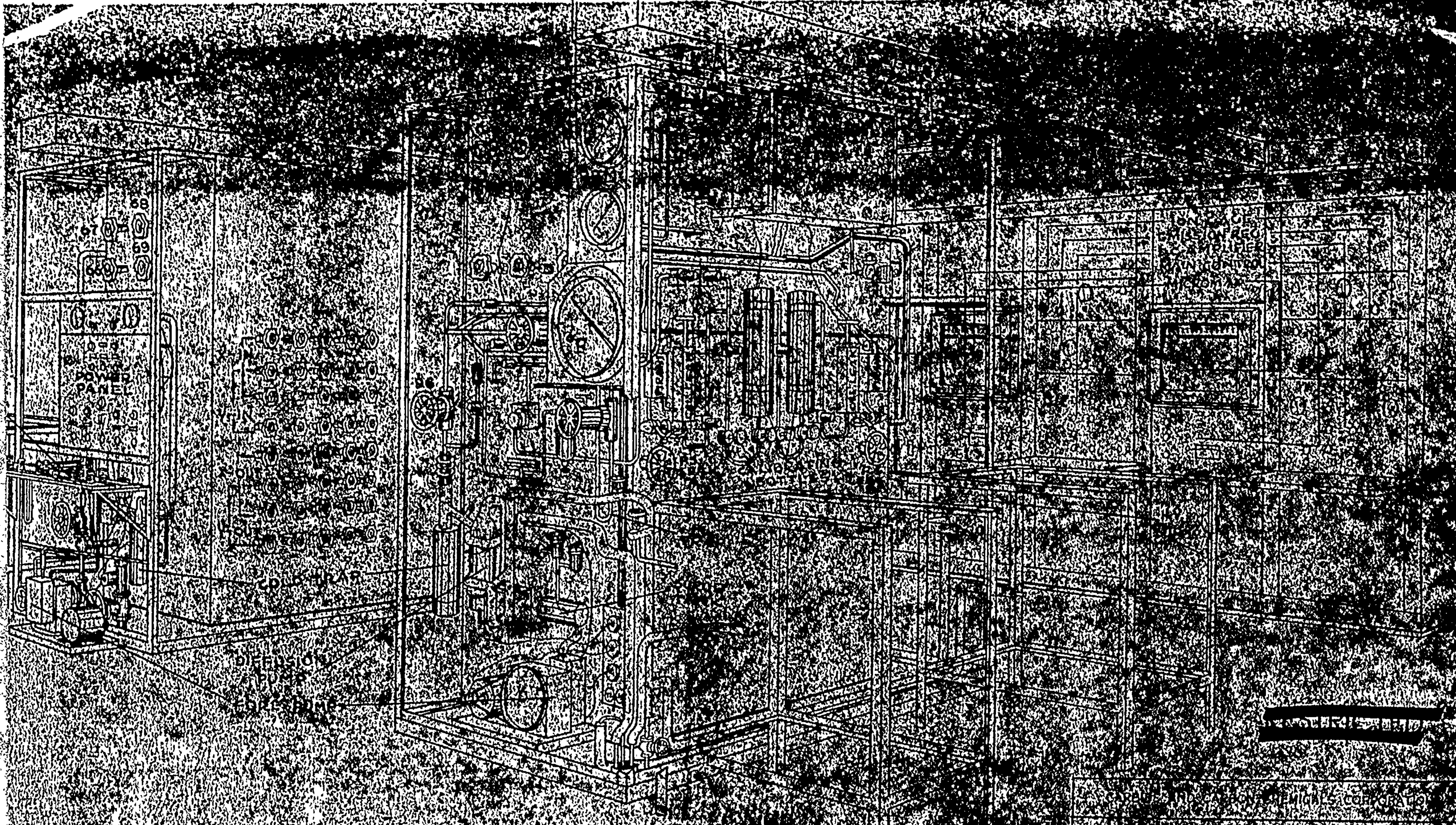
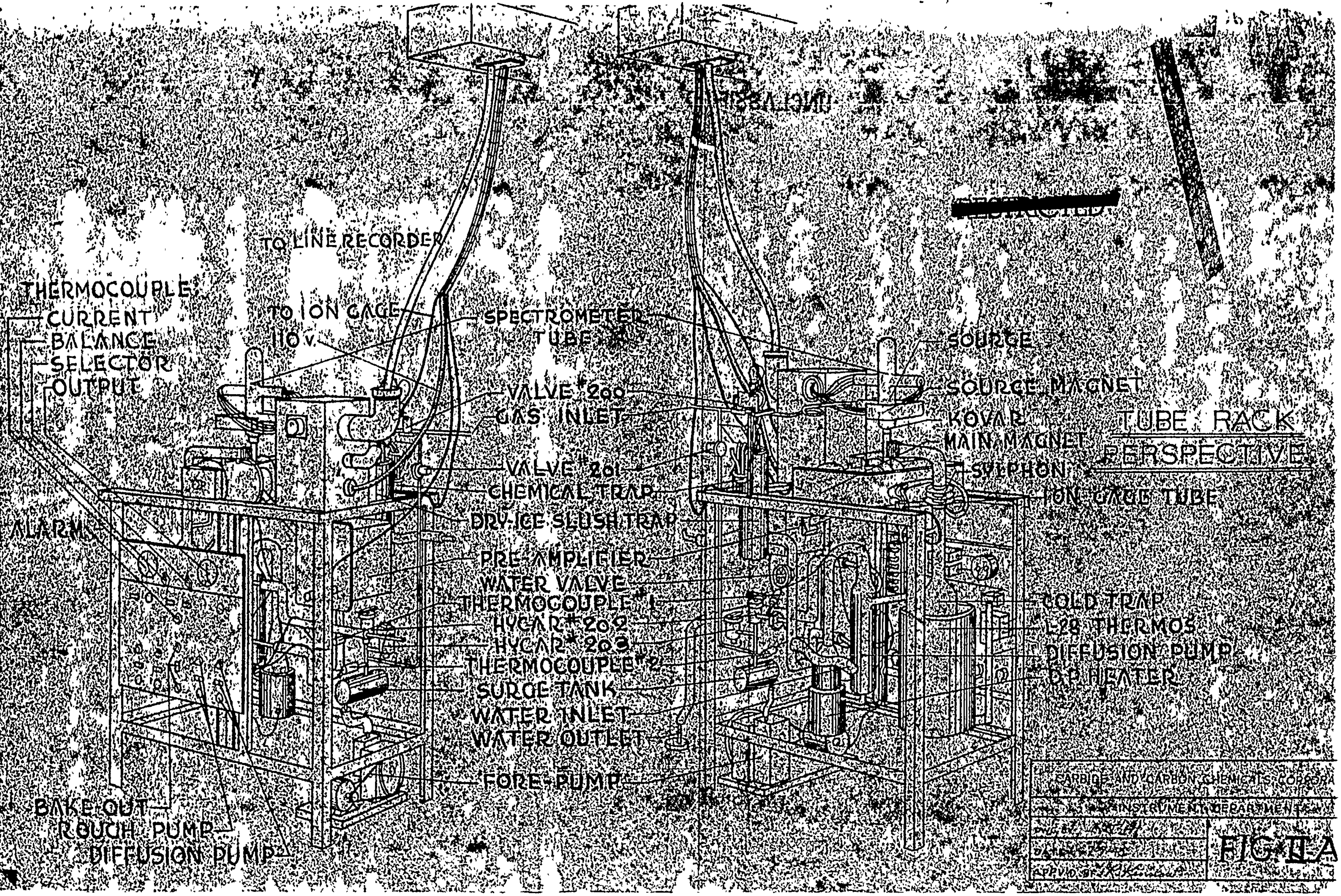


MANIFOLD - PERSPECTIVE

FIG II A



TUBE RACK PERSPECTIVE

FIG IIA

TABLE OF CONTENTS

[redacted]

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INTRODUCTION

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A. Purpose of Manual

This Operations Manual has been prepared as a guide to efficient operation of a Line Recorder Station. It is hoped that the manual will aid in achieving uniform operating procedure throughout the department.

Although this manual is intended to be of most benefit to the station operator, it is felt that the entire personnel of the Line Recorder Department will gain useful information from this work.

B. Scope of Manual

Included in this manual is a brief discussion of the theory of Line Recorder operation, a description of the equipment used, the function of the Central Control Room, and sections covering routine, non-routine, and emergency procedures to be followed in the operation of a Line Recorder Station.

The material and procedures described herewith are those which conform with the best information available at the present time. However, it should be understood that the various sections of the manual are subject to revision at any time.

[redacted]

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C. Principles of Line Recorder Operation

The Line Recorder is an instrument which is used to determine the composition of the process gas being treated in the 300 section. It employs a Mass Spectrometer and a Micromax Recorder in order to indicate and record continuously the changes in this composition.

The principal functions of the Line Recorder are to determine whether or not the leakage of G-74, air, or C-816 into the C-616 is normal, and to assist in locating an abnormal leak in the system. This is essential in order that the leaking equipment be separated from the rest of the Cascade so that normal operation may be continued.

The Mass Spectrometer is based on the principle that gas particles, which have been passed through an electron beam and have been converted into positive ions, will move from a higher to a lower potential in a definite path through an electrical field.

A magnet can bend this ion beam since the charged particles tend to move along a curved path in a magnetic field. The different types of particles can be sorted out since the heaviest ions are least affected by the magnetic field while the lightest particles are deflected the most.

[redacted]

those ions of a cer[illegible] amount proceed to a collector plate. By measuring the current required to convert the ions back to uncharged particles, an indication of the amount of that component present in the M. S. Tube is obtained. To measure the amount of another component, the speed of the ions is changed by varying the acceleration voltage so that the desired ion beam can be focused directly on the collector plate. The lighter particles require a greater acceleration voltage so that they travel at a higher speed and are not over-deflected by the magnet.

The Line Recorders are located in stations on the operating floor, each station serving two buildings and containing two recorders for each building. By means of a manifold, a small continuous stream of process gass from the 6A pump of the cell being analyzed can be circulated past an adjustable leak. A small fraction of the sample stream is bled through this leak to be analyzed by the Mass Spectrometer; the remainder of the sample is returned to the 6A suction of that cell. Samples may also be withdrawn from the intersectional cells and returned to the suction of an appropriate "A" pump.

One recorder of each building is usually connected to the top active cell of that building.

[redacted]

It is called the "principal recorder" and its record is simultaneously reproduced on a "slave recorder" in the Central Control Room. The other recorder is the "spare recorder" and it can be used for scanning to check the compositions in the other cells of the building or as a check on the "principal recorder".

The C-616 is removed from the sample stream before it reaches the Mass Spectrometer tube since it would cause damage to the tube. The percentage of C-616 can be estimated by subtracting the sum of all measured impurities from the total sample flow. The flow to the M. S. tube must be kept constant to provide significant readings.

The function of each part of the Line Recorder unit to provide continuous gas analysis will be covered in more detail in the following section.

D. <u>Description of Equipment</u>

1. MANIFOLD:

The Line Recorder Manifold is a piping system which serves several purposes in L. R. operation. Different sections of the manifold provide means for:

a. Maintaining a flow of process gas from the 6A discharge of any cell in that building to the tube rack for analysis by the Mass Spectrometer unit.

[redacted]

b. Evacuating all manifold lines that may contain process gas and for purging these lines with G-74 if necessary.

c. Removing a PG sample from any cell for laboratory analysis.

d. Static and dynamic calibration of the M.S. unit and for periodic checks of this calibration.

Process gas is withdrawn from the 6A discharge of a cell through a 5/8-inch copper line. It passes to the pipe gallery to block valves set beneath each station. The gas flows to the manifold and into either the incoming top or bottom header, then through the various valves and past the adjustable leak before returning to the outgoing top or bottom header and back to the 6A suction of that cell. A diagram of the L. P. Manifold is included as Fig. 1.

In the manifold, flow from each cell is blocked off by closing the Crane "T" valves which continue to permit flow through the loop. If cell 10 is being analyzed by Tube Pack "B" on the Lower Loop, it can be seen from the diagram that the following valves should be open: "T" valve 10 on the Incoming Bottom Header, 65, 66, and "T" valve 10 on the Outgoing Bottom Header. "T" valves 58, 64, 63, and 69 are kept closed, and the flow is then confined to the main line.

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To withdraw a small portion of this flow for analysis, adjustable leak 71 is cracked slightly until the proper flow rate (as indicated by the Pirani Gage) is achieved. Opening valve 76 admits the gas to Tube Rack "B".

All valves and lines for the aforementioned flow are incased in a metal enclosure in which dry air is continually circulated. The proper temperature is maintained by Calrod heaters controlled automatically.

The evacuation system consists of a mechanical pump and a diffusion pump connected to suitably valved lines so that any portion of the manifold can be cleared of PG or contaminants.

The proper valves to be opened for evacuating various parts of the manifold can be determined from the diagram. Further discussion on pumping-down procedures is included later in this manual.

Samples of C-616 may be withdrawn for analysis in the laboratories by condensing the C-616 in a cold trap, pumping off the non-condensable gases, and vaporizing the C-616 into a sample tube.

[redacted]

Samples may be collected and sealed in a simple U-tube, but the Hoke tube is now in common use.

The sampling equipment consists of two sampling manifolds by which the process gas circulating through the manifold can be directed into either a U-tube or a Hoke tube. The diagram indicates the valve arrangement on the Hoke tube sampling manifold. The sample is drawn off through either valve 68 or 69 through 90-1 and is transferred from the cold trap to the Hoke sample tube as described on page 121. The U-tube manifold ~~(not indicated on the diagram~~) is similar. A mechanical pump is provided for evacuating the sampling manifold and a thermocouple gage indicates when the desired evacuation has been reached.

The static calibration system is used to admit samples of known composition into the M.S. tube so that the proper constants can be determined to convert signals into actual percentages. This calibration is used when a Line Recorder is first put into operation or after major repairs or changes in equipment have been made.

The calibration equipment consists of 2 two-liter cylinders which are filled with gas mixtures of known concentration, and suitably valved

[redacted]

lines leading to the calibration leak [illegible] the gas sample flows into the M.S. tube.

The static calibration is checked periodically by means of the <u>dynamic check</u>. This consists of admitting a small measured volume of ambient air from the manifold casing directly into the PG stream being analyzed. The air is passed through a rotameter to determine its flow rate. The PG flow is measured with a Venturi flow meter before being mixed with the air. Readings are taken on the Micromax before air is admitted and these readings are compared with those obtained from a sample of known PG-air composition to check the calibration of the instrument.

The static calibration may also be checked by admitting 100 per cent air from the manifold casing directly into the M.S. tube through the adjustable calibration leak. Another common check is to use a 95% C-616, 5% G-74 sample in a calibration cylinder and admit this mixture into the M.S. tube as in the Static Calibration procedure. For further information on these checks, see pp. 109 to 117.

~~CONFIDENTIAL~~

2. <u>Tube Rack</u>

The tube rack is an assembly of the various elements of a mass-analyzing system. This assembly is best described by following the flow through these various elements including a brief description of each. (See Fig. 2)

The sample of gas to be analyzed is introduced at the tube rack inlet and flows through the <u>chemical trap</u> (151) into the Mass Spectrometer Tube. The chemical trap is necessary for the removal of C-616 from the sample stream before it reaches the M.S. tube; otherwise, it would greatly shorten the life of the tube.

The chemical trap consists of a vessel of mercury, whose vapor difusses up to the cold spot where it reacts with C-616. The cold spot is cooled by a copper finger submerged into a slush trap of trichlorethylene and dry ice. A warming fin is attached to prevent the mercury vapor from condensing before it reaches the cold spot.

The gas sample is passed into the <u>M.S. tube</u> (150) directly at the ion source. The source end is housed in a glass envelope and is that part of the tube in which the gas particles are converted into

[redacted]

positive ions.

The filament in the source emits an electron beam which is caught by the plate. The electron beam is kept aligned by a magnetic field which is obtained by means of an Alnico magnet surrounding the tube. The ions formed by collision of the electrons with the gas are accelerated through a series of plates which align and increase the velocity of the ion beam. This ion beam enters the copper analyzer tube and passes between magnet poles. The different types of ions, while in the magnetic field, will travel in arcs of different radii which depend upon the mass, energy and charge on the particle.

The accelerating voltage on the plates is varied to focus the individual ion "peaks" on the collector plate, where an ion current is produced and is indicative of the amount of that component in the M.S. tube. The ion currents may be as small as 10^{-12} amperes and must be amplified to be measurable. The <u>pre-amplifier</u> located on the tube rack partially accomplishes this important function.

The <u>diffusion pump</u> (159) is employed to maintain the high degree of vacuum required for the successful operation of the M.S. tube, namely 10^{-5} to 10^{-7} millimeters of mercury. The <u>Ion gage</u> (160) is an electronic method of measuring the low pressure on the suction side of the diffusion pump by determining the amount of ionization produced by electrons.

 [redacted]

Positive ions collected at the plate produce a current which can be measured and this current is proportional to the pressure in the gage. In order to evacuate the M.S. tube successfully the diffusion pump must be "backed" by a fore pump.

The fore pump is a mechanical pump, the function of which is to evacuate the system to a pressure below 0.1 millimeter of mercury and to maintain this or a lower pressure at the exhaust of the diffusion pump.

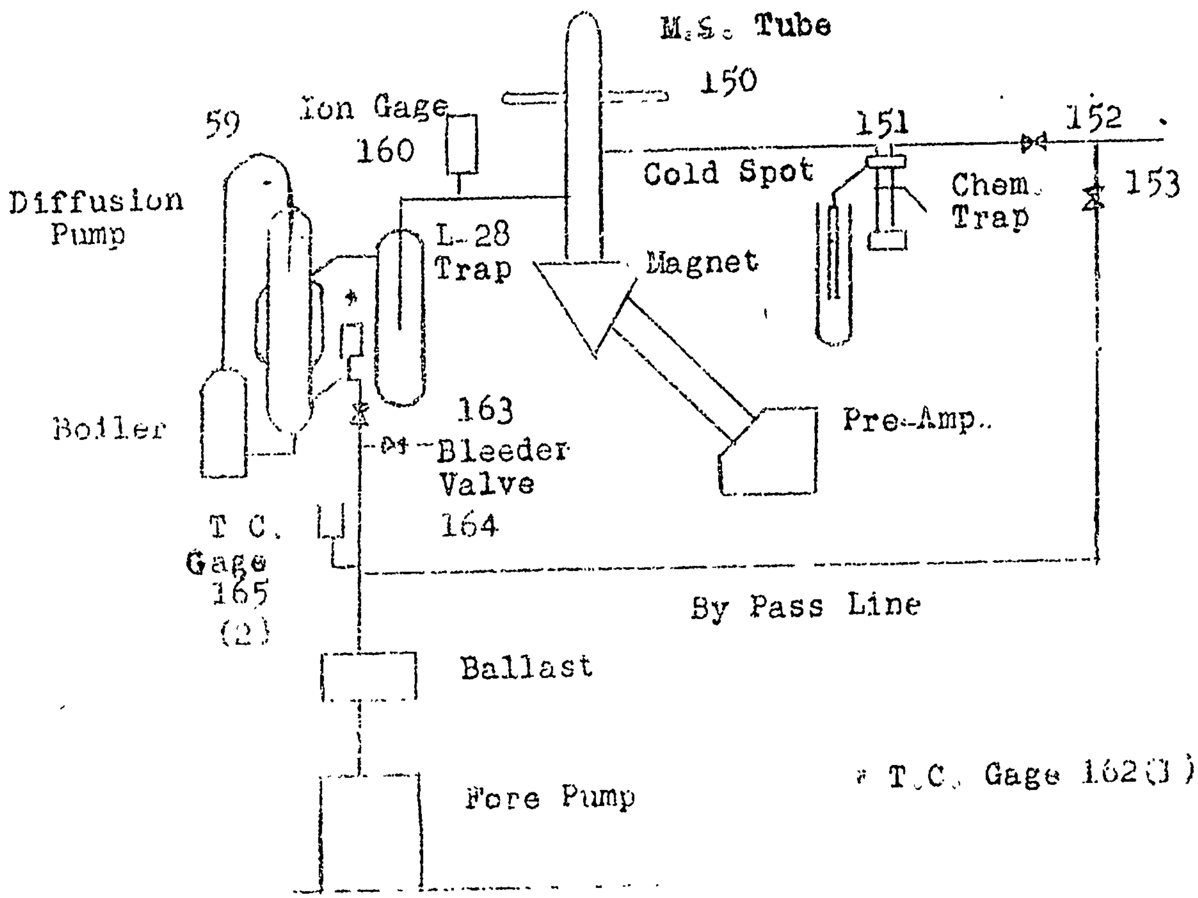
Just upstream of the fore pump is located a ballast tank, or oil trap. This is included so that, in the event of a fore pump failure, the oil from that pump will not be drawn into the diffusion pump. Valve (164) is provided so that the system may be vented to the atmosphere. Valve (163) is provided so that the diffusion pump may be isolated when the fore pump is used for other pumping or must be replaced. Thermocouple gages (162) and (165) measure the fore pump vacuum at the diffusion pump and the fore pump respectively.

Valve (153) is incorporated so that after making changes the fore pump can be used to pump down the gas lead to the tube rack before opening valve (152) to the spectrometer.

[redacted]

TUBE RACK

Figure 2



Relay Rack

The relay rack assembly is a bank of panels housing the various electronic units necessary for the proper functioning of the mass-analyzing system. In a typical station, this bank consists of a main power panel and 4 identical Line Recorder units of two panels each.

The main power panel consists of 12 switches which control the supply of electric power to the Line Recorder units, tube racks, manifolds and necessary auxiliary equipment. The panel is divided so that all switches on side control the units of one building and those on the other half the adjoining building. Considering the switches for one building, their functions are as follows:

1 - The top switch is connected to the main power line and supplies power to the remaining switches.

2 - The next lower switches control the power to the Line Recorder units. The left switch for the B unit and the right for the A unit.

3 - The next lower switches control the power to the tube racks with positions similar to those for the L. R. units.

4 - The lowest switch controls the power to the manifold and auxiliary sampling equipment.

[redacted]

The Line Recorder panel units house the following electronic circuits with their corresponding panels:

1 - Voltage Stabilizer
2 - High Voltage Supply
3 - Main Control Panel
4 - Emission Regulator
5 - D. C. Amplifier
6 - Micromax
7 - Ion Gage

A brief description of the function of each unit and of the component parts follows:

1. <u>Voltage Stabilizer</u>

The voltage stabilizer tends to reduce fluctuations in the 110-volt supply. This "stabilized" voltage is then supplied to the other electronic units; thus the design of these units is simplified since less voltage regulation is necessary. No controls are provided for the voltage stabilizer so a special panel is not included.

2. <u>High Voltage Supply</u>

The function of the high voltage supply is to furnish the main control panel with 2500 volts D.C. It converts the 110-volt A.C. output of the voltage stabilizer into 2500 volts D.C.

The milliammeter provided on this panel is used to check the operation of the regulator in the High Voltage Supply. The red pilot light indicates that this unit is operating.

[redacted]

Main Control Panel

The function of the Main Control panel is to supply the proper accelerating voltage at the source of the M.S. tube and a potential at J_5 which is varied according to the mass being analyzed.

The 2500 volts is provided from the high voltage supply and is applied through a variable resistance (trimmer) to the voltage dividers. One divider is used for manual control to supply the desired potential to the J_5 plate. With the Manual Divider, the applied voltage can be varied from zero to the maximum obtainable voltage.

The other divider consists of a series of potentiometers and fixed resistance. Each potentiometer has a center-tap which is moved by means of a screwdriver adjustment. By this means the potential of J_5 can be selected from one of the center-tapped potentiometers and applied to measure a certain mass as with the manual divider. Each of these screwdriver adjustments, are for a certain range of voltages which are used to direct the all ions of a certain range of masses towards the collector plate. Hence each adjustment has been assigned to a certain channel with a restricted range and is labelled from A to H.

[redacted]

The use of the Manual Divider or the channels with certain screwdriver settings is governed by the setting of the Accelerating Voltage Selector switch. On "Manual", the manual divider control is used; on "Point Check", the mass designated by the Point Selector switch is analyzed; on "Record", normal operation is provided where the channel selected by the program of the Micromax Recorder provide the accelerating voltage at any one time.

The Trimmer enables the operator to adjust the machine for small voltage drifts which cause readings to be "off peak". By a combination of the proper trimmer setting and screwdriver adjustments, the desired masses can be analyzed according to a specific program.

4. Emission Regulator

The function of this electronic circuit is to maintain a constant emission of electrons from the M.S. tube filament. This is done by holding the voltage across the filament wire constant even though the resistance varies with changing conditions.

The emission regulator also provides a stabilized voltage supply for the J_1, J_2, and J_3 plates and the trap of the ion source box. These voltages are maintained constant with a voltage regulator and are set with the J_1 & J_2 coarse, J_1 Fine and J_3 Focus Control switches. When properly set, the emission current as indicated on the microammeter should remain constant. Fluctuations in this current indicate poor regulation.

The Power switch turns on the current so that the tubes may warm up. The Filament switch controls the power to the M.S. tube filament. The Regulate and Emmission Coarse and Fine knobs are used to vary the electron emission of the M.S. tube filament. The Instrument switch provides a means of reading large emission or trap current values on the microammeter.

The A.C. Ammeter indicates the current being used to heat the filament. This reading decreases over a long period of time and the ammeter gives an indication of the filament condition. The Focus knobs are used to vary the various potentials on the focusing plates so that the ion beam is properly directed towards the collector plate.

5. D. C. Amplifier

After the gas admitted to the M.S. tube has been ionized the ions are directed to the collector plate where they are neutralized by a flow of electrons.

[redacted]

This electron flow passes through a resistor and a small potential drop is thereby created. It is the function of the D.C. Amplifier to amplify this small effect to a current which is measurable on the Output meter or directly on the Micromax Recorder.

From the small voltage drop of the resistor, currents below 10^{-12} amperes may be amplified by the pre-amplifier unit (located on the tube rack) and sent to the D.C. Amplifier where further amplification occurs. The current is thus brought up to about 1/2 milliampere and is therefore measurable by the Output meter or the Recorder. Sensitivity controls are provided so that the meter does not read off-scale with widely varying ion currents. Likewise, sensitivity controls for each of the 8 channels are provided to prevent off-scale readings of the recorder. The sensitivity decreases as the Sensitivity Factor increases, since the actual amplifier output, or signal, is the product of the reading and the sensitivity factor.

The switches labelled "Fil"", "Reg", and "Battery Check' refer to the filament emission, regulation, and battery voltage. These should be checked as described on page 136 for proper operation of the D.C. Amplifier.

[redacted]

The Coarse, Medium, and Fine Balance adjustments are provided to counterbalance the drifts in the minimum output current. Thus the amplifier zero can be set so that variations in the signals without actual changes in concentration will not occur.

6. Micromax Recorder

The recorder is an automatic self-balancing, 16-point recording potentiometer. It serves as the "brain" of the Line Recorder unit and has two chief functions:

1 - According to a pre-arranged program, the recorder selects the high voltage necessary to measure ions of a certain mass and this voltage is supplied at the M.S. tube source through the Main Control panel.

2 - The recorder receives the output of the D.C. Amplifier and prints this signal on a chart with the proper symbol.

During one cycle of 6.4 minutes, the channels can be scanned in any desired manner to give a complete program of 16 points. A typical program is arranged so that the mass 14 signal prints 8 times, the Pirani reading twice, and the amplifier zero and masses 20, 28, 32, 44 and 69 once each per cycle. Between any two printings, the time elapsed is 24 seconds.

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The recorder employs a synchronous motor and a Wheatstone bridge circuit to actuate a print wheel which traverses the strip chart horizontally in either direction. The strip chart moves at the rate of 3 inches per hour or one horizontal line every ten minutes. The chart is divided into 100 divisions, from -10 to + 90.

The switch for the motor is located at the top of the recorder cabinet. Further discussion of the Recorder is given on page 140.

7. Ion Gage

The ion gage unit serves to indicate the pressure in the M.S. tube to ascertain whether the pressure is sufficiently low for safe operation. The electronic circuit also provides a safety cut-out arrangement whereby the current supplies to both the M.S. tube and ion gage filaments are tripped when the pressure in the tubes exceeds 5×10^{-5} mm Hg. Thus the danger of burning out the filaments during a loss in vacuum is greatly reduced.

The ion gage is a means for measuring very low pressures. The current necessary to neutralize ions formed in the ion gage tube is a measure of the pressure in that tube. This current is amplified and the output is measured with a meter scaled to read directly in mm of mercury. A

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sensitivity switch permits measuring pressures from 1 x 10^{-4} to below 1 x 10^{-7} mm mercury.

The various controls on the ion gage panel are for checking the operation of this unit and for making the adjustments necessary to maintain proper operating conditions. A more complete description of these controls is given under Ion Gage Operation on page 137.

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E. Central Control Room

The Central Control Room is concerned primarily with the prompt detection of any abnormal inleakage of contaminants into the Cascade equipment and with giving advice regarding the steps necessary to correct the situation.

To accomplish this important task effectively, it is essential that the personnel in the C.C.R. be kept thoroughly informed of Process and Line Recorder operations. This information is relayed to a system of "slave recorders" in the Central Control Room to provide a continuous record of the analysis of the plant stream at one point in each building. These recorders are connected to the "principal recorders" of each station which can be set to analyze the proper cell in accordance with the needs of C.C.R.

Information can also be relayed to C.C.R. via the P.A.X. phone system (Nos. 23, 24, or 25) or via the Bell System phone (No. 8-9457). Thus, communication with all Station operators and supervisors, building foremen, Cascade Coordinators, 600 section, etc. can be maintained.

Records of process changes (cell history, feed rate and conditions, purge rate, 600 section operation, etc.) must be kept up-to-date. In addition, routine information regarding changes in building and Station operation (such as variations

~~CONFIDENTIAL~~

from normal cell operation, changes in sensitivity settings, or in the operation of both the "principal" and the "spare recorders") should be made available to C.C.R. to enable the proper interpretation of the Micromax charts.

Since the Control Room provides the best overall picture of Cascade operation, it is essential to work with C.C.R. when Cascade emergencies arise. Although operations such as scanning and checking inleakage rates are done in the Station, it is frequently possible for the C.C.R. to assist materially by advising the Station operator regarding the best and quickest procedure. C.C.R. will also relay information to the Stations when process changes which will affect Recorder readings are expected. This is especially true since it is not possible at present to set up any hard-and-fast rules to cover every possible emergency.

To keep all "principal recorders" operating properly to be ready for any emergency, it is necessary for the C.C.R. personnel to watch the operation of this machine and to advise when changes should be made. Thus, the Station operator and the C.C.R. should work in close cooperation for the mutual benefit of both.

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ROUTINE PROCEDURES

A. Manifold Operations

1. Changing Sample Point

It is frequently necessary to change from the analysis of one cell to that of another. The proper procedure is:

1 - Close inlet valve of cell being analyzed.

2 - Close outlet valve of same cell.

3 - Open outlet valve of cell to be analyzed.*

4 - Open inlet valve of new cell.

5 - If necessary, vary the adjustable leak to obtain the proper flow to the M.S. tube. (See pp. 102-108)

6 - Allow a few minutes to elapse before assuming that the Micromax readings are truly representative of the composition in the cell.

When closing these manifold valves, always torque them to approximately 30 foot-pounds using the torque wrench. Consult shift supervisor regarding the reading of the dial on the torque wrench.

When opening valves, open it wide and then close it 1/4 turn in order to facilitate checking the setting of a valve.

---

* If the new cell is an intersectional cell, open the outlet valve of the nearest onstream cell in that building which is not being analyzed.

[redacted]

The Tejax indicators are used to indicate the valve setting. When the pointers are in the "12 o'clock position", the valve is closed; any other setting indicates an open valve. However, these Tejax indicators are unreliable since "sticking" often causes the readings to be off. Therefore, it is advisable to check valve settings manually rather than rely on the indicators.

2. Adjusting Sample Flow Rate

The operation commonly referred to as "adjusting the Pirani" is that of regulating the adjustable leak so that the proper flow into the M.S. tube may be maintained. This is important for several reasons:

1 - The instrument is calibrated using a certain flow rate; any other rate of flow will send too much or too little of the measured components into the M.S. tube and incorrect readings will result.

2 - A high rate of flow may endanger the M.S. tube since the rate at which the C-616 is removed in the chemical trap may be exceeded.

3 - A high flow rate may result in loss of vacuum with the danger of tripping off the filaments. It is also desirable to remove a very small amount of PG from the manifold for analysis because of the valve

or this material.

Although the Pirani gage is actually a device for measuring low pressures, it is used here to indicate the sample flow rate since the flow is proportional to the pressure under the conditions used. It is important to keep the bridge current at the proper value since small changes in this variable cause large deviations in the Pirani reading.

The adjustable leak is normally set to give a Pirani reading of 50 plus or minus one. However, if the gas concentration differs appreciably from the normal low percentage of contaminants, a correction must be applied to the above value to give the proper flow rate. This is further discussed on page 106.

The following are possible explanations for difficulty in maintaining the desired Pirani reading:

1 - No two adjustable leaks are the same. they differ in <u>sensitivity</u> and in amount of "<u>free play</u>". The less sensitive leaks must be turned more to produce a certain change in Pirani reading than the sensitive ones. Those with "free play" can be turned slightly with no actual change in flow rate. These should be adjusted so that all slack is taken up and the Micromax readings will then show less "scatter".

[redacted]

2 - It is often easier to adjust the leak by approaching the proper setting from one direction only. Closing the valve slowly has been found to give better control.

3 - Check the bridge current frequently since any change from the value set during calibration will cause the Pirani to read incorrectly.

4 - The Pirani should never read higher than "60" or the chemical trap is in danger of being overloaded. A high Pirani reading may be due to any of the following:

<u>Surges</u>, or temporary pressure fluctuations in process, often occur. These may often be ascertained with the cell panel board.

<u>Changing cells</u> may change the Pirani reading since the 6A discharge pressure varies from cell to cell in a building. Cell 2, normally the top active cell, usually has the highest discharge pressure because of the added resistance to flow provided by the building by-pass lines. Cells on Inverse Recycle often are at higher pressures than onstream cells because of the continuous addition of G-74.

<u>Concentration changes</u> affect the Pirani since the average molecular weight is a factor which has an effect on the flow rate. High G-74 concentrations will send the flow rate up and the Pirani reading may go offscale. This sends a "slug" of gas

[redacted]

into the 1S base with the danger of raising the pressure to the point where the filament trips off. It is therefore important to know the approximate concentration of the cell to be analyzed and to take the necessary precautions. This is particularly true in the top building of the Cascade or in changing to a cell on Inverse Recycle or Direct Recycle.

<u>Leakage</u> from the seals or in the cell will affect the Pirani reading if the pressure or concentration differ from normal. The same is true for manifold inleakage.

5 - Never close or open the adjustable leak more than "fairly tight" with the hands. Damage to this part may otherwise result.

6 - Experience has shown that the Pirani reading of one machine will rise if the other machine is placed on the same cell. This may be due to an increased pressure in the manifold loop above the adjustable leak because of a rise in the 6A <u>suction pressure</u>. A small adjustment in the leak setting should therefore be made.

If there is undue difficulty in obtaining the correct Pirani reading, contact your supervisor.

The <u>Pirani Setting Curves</u> are used to determine the proper Pirani reading for which the adjustable leak should be set for different compositions of gas being analyzed.

[redacted]

The correct Pirani setting is determined using the signals from mass 69 (C-816), mass 14 (G-74), and mass 32 (oxygen). The proper setting is obtained in the following manner:

1 - Adjust the leak to give a Pirani reading fairly close to 50 (between 45 and 55) and obtain the 69, 14, and 32 signals.

2 - Using the 69 signal obtained, read the proper Pirani setting from the top graph (see graph 1 on following page).

3 - Using the 14 signal, determine (from graph 2) the correction for G-74 and apply this to the setting obtained in <u>step 2</u>.

4 - Using the 32 signal, determine (from graph 3) the correction for oxygen and apply this to the setting obtained in <u>step 3</u>.

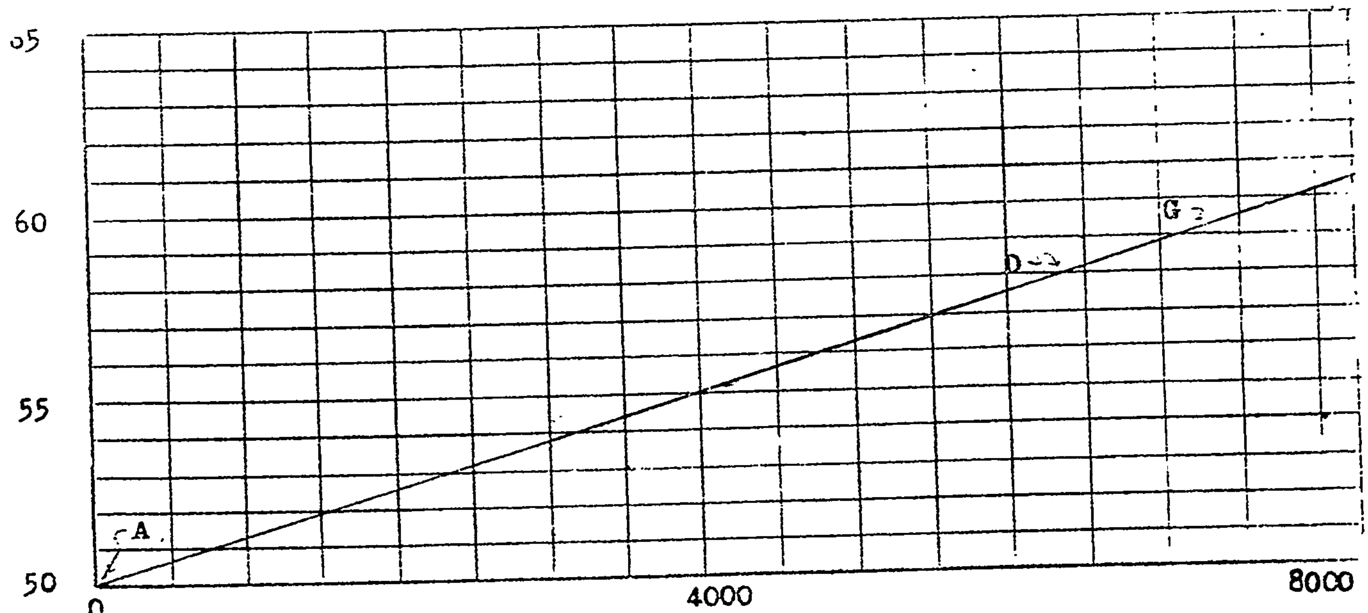
5 - Set the adjustable leak to give a Pirani reading as determined from <u>step 4</u>. Obtain a new set of 69, 14, and 32 signals.

6 - If the Pirani setting was changed appreciably from <u>step 1</u>, it may be necessary to repeat <u>steps 2</u> through 5 to determine the proper pirani reading.

For illustration, two examples will be given. The first example assumes low concentrations of C-816, G-74, and oxygen while the second assumes high concentrations. The graphs on page <u>107</u> are typical of those actually provided in the Station for use by the operator.

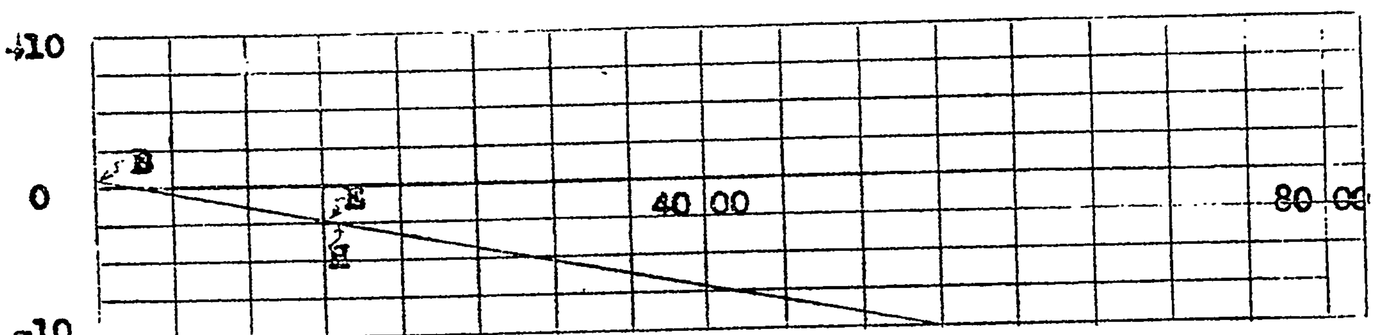
[redacted]

Pirani Setting Curves



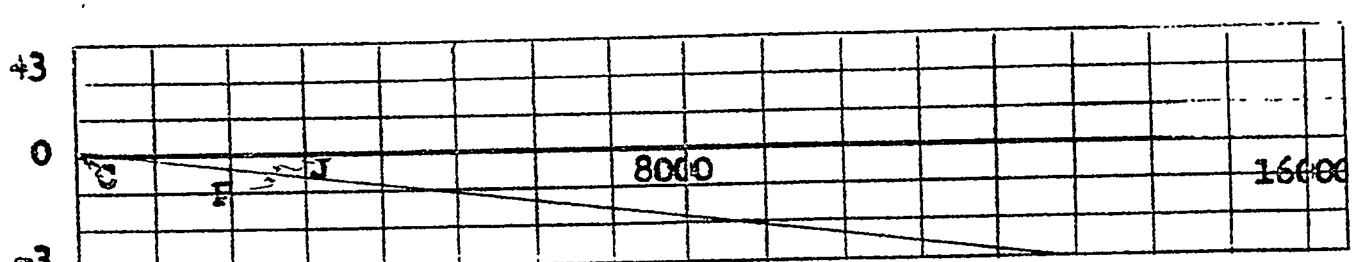
GRAPH 1

Mass 69 Signal



GRAPH 2

Mass 14 Signal



GRAPH 3

Mass 32 Signal

[redacted]

Example 1

Pirani reads $49\frac{1}{2}$, 69 signal is 22. 14 is 1 and 32 is 2. From Graph 1 (Point A), it is seen that the Pirani setting is approximately 50. The G-74 correction is found from Graph 2 (Point B) to be approximately $+\frac{1}{2}$, so the Pirani setting should be $50\frac{1}{2}$. The oxygen correction is zero (Point C), so the corrected Pirani reading remains $50\frac{1}{2}$. Upon adjusting the leak to give a Pirani reading of $50\frac{1}{2}$, it is found that the 69 signal rises to 23 and the G-74 and oxygen signals are unchanged. Therefore, the proper Pirani setting is still $50\frac{1}{2}$.

Example 2

The Pirani reading is $50\frac{1}{2}$, mass 69 reads 63 with a sensitivity factor of X 100 (or 69 signal is 6300), and the G-74 and oxygen signals are 1500 and 2500 respectively.

From Graph 1 (Point D), the Pirani setting is 58 for a 69 signal of 6300. The G-74 correction (Point E) is $-2\frac{1}{2}$ and the oxygen correction (Point F) is $-\frac{1}{2}$ so the proper Pirani setting is $58-2\frac{1}{2}-\frac{1}{2}$ or 55. The leak is then opened to give a Pirani reading of 55.

The new signals are: 69, 7250; 14, 1600; and 32, 2700. Using the graphs, it is seen that 59.2 is the Pirani setting for the 69 signal (Point G), and a correction of $-2\frac{1}{2}$ (Point H) results from the G-74 signal with a correction of $-\frac{1}{2}$ (Point J) for the oxygen signal. The corrected Pirani setting is then 56.2.

[redacted]

Upon readjusting the leak to give this Pirani reading, and checking the new signals on the chart, it is found that this is the proper setting.

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Extrapolation of Recorder Readings When The Pirani Is Low

Certain buildings and sections of the [illegible] are operating at reduced pressure of appx. [illegible] psia and because the adjustable leaks have not been designed to work at these low pressures, the flow of gas will not rise to a Pirani reading of "50".

In such cases as this, be careful not to open the leak so far that the pin will be sheared, but open it until the maximum Pirani reading is obtained. Then when the correct percentage readings are to be obtained use the equation below:

$$Sm \text{ (in \%)} = \frac{50 \text{ (Signal x Sensitivity X Calibration Constant)}}{\text{Pirani Reading}}$$

NOTE: The readings obtained from this equation are accurate to within ±5%, providing the G-74 concentration in the PG System is 5% or less.

[redacted]

3. SPECIAL OPERATIONS

A. Static Calibration

(For Operator's information only - This Procedure is employed by the Maintenance Dept.)

To calibrate a Line Recorder, three samples of gases are needed in the order listed below:

1. 5% G-74, 95% C-616
2. 100% Air
3. 5% G-74, 2% G-816, 93% C-616

PROCEDURE:

1 - With the Line Recorder working properly (L.R. #A) open valves 92, 91, 81, 82, 84, 96, and 97. Pump the lines down to approximately 5-10 microns and then close valve 91.

2 - Record residuals that are printed on the Micromax strip chart.

3 - Admit sample #1 (5% G-74, 95% C-616) by opening either valves 77 & 79 or 78 & 80 depending upon which valves are attached to the #1 Sample Bottle. This shall be done by first opening valve 80 (assuming that the lower bottle contains the number one sample) and then cracking valve 78 and immediately closing it again.

4 - Regulate the Adjustable Leak until the Pirani will read "50". If this value can not be obtained, again crack valve 78 until the flow will register "50" on the Pirani Gage.

 [redacted]

5 - Adjust emission of the M. S. Filament until:

$\Delta S_{14} = 50$ (% of G-74 in Sample #1)

Where ΔS_{14} is total S_{14} minus the residual S_{14}.

6 - Record emission and trap current.

7 - Record all peaks.

8 - Pump out Sample #1 by opening valve 91 and allowing the Pumping equipment to bring the pressure down to 5-10 microns, then close 91.

9 - Record residuals of all peaks.

10 - Admit sample #2 by closing valve 96 and opening valves 91 & 100. 100 is cracked and then immediately closed and the Adjustable Leak is regulated so that:

$\Delta S_{14} = 3900$

Note: Emission is held constant at the value determined in Procedure #5.

11 - Record Pirani Reading and all Peaks.

12 - Pump out Sample #2 by opening 96 and then follow Procedure #8.

13 - Record all residuals.

14 - Admit sample #3 by opening valves 77 & 79. Valve 77 is cracked and then immediately closed. Regulate the Adjustable Leaks so that:

$\Delta S_{14} = 50$ (% of G-74 in Sample #3)

15 - Record Pirani reading and all peaks.

[redacted]

[redacted]

16 - Plot a Pirani correction curve for nitrogen in the way shown: at "0" correction (on the "Y" axis) place a point at S_{14} (the "X" axis) obtained in procedure #5. At a signal of 3900 place a point at a Pirani Correction equal to (0.78 x (Pirani reading of #11 - 50). Now draw a straight line through these two points.

17 - Plot a Pirani correction curve for OXYGEN in the manner described below: At "0" correction and zero signal place a point. At S_{32} obtained in #11 place a point at the Pirani correction equal to 0.21 x (Pirani reading of #11 - 50), and draw a straight line from this point to the one obtained above.

18 - A Pirani setting curve is needed for C-816. This is obtained by placing a point at a Pirani setting of 50 and a signal of zero. From the reading obtained in #15 (Sample #3) obtain the G-74 signal. From this and the curve plotted in #16 the Pirani correction due to G-74 is obtained. This result is then substracted from the Pirani reading obtained in #15 and the remainder plotted against the S_{69} that was obtained in #15 procedure and the two points are connected with a straight line.

19 - The Calibration Constant for the different Signals is obtained as follows:

Constant for S_{14} = 0.02

" " $S_{28} = \dfrac{\text{(\% of G-74 in Sample \#1)}}{S_{28} \text{ from \#7 procedure}}$

" " $S_{32} = \dfrac{\text{(\% of Air in Sample \#2) x 0.21}}{S_{32} \text{ from \#11 procedure}}$

" " $S_{69} = \dfrac{\text{(\% C-816 in Sample \#3)}}{S_{69} \text{ from \#15 procedure}}$

Then,

$$\% \text{ G-74} = 0.02 \times S_{14} \times \frac{\text{calculated Pirani reading}}{\text{actual Pirani reading}}$$

$$= \text{Constant } S_{28} \times S_{28} \times (\text{" " " "})$$

$$\% \ O_2 = \text{Constant } S_{32} \times S_{32} \times (\text{" " " "})$$

$$\% \text{ C-816} = \text{Constant } S_{69} \times S_{69} \times (\text{" " " "})$$

B. Dynamic Check:

This system of calibration is to be used frequently for a quick calibration check on the Line Recorder. The method used is to admit a sample of dry air into the manifold, and from the readings of a flowmeter and a rotameter, the percentage of air admitted into the stream in relation to the PG Stream flow may be calculated. This increase in air percentage causes an increase in the G-74 concentration, which is measured by the Line Recorder. The increased reading on the Micromax should then equal to the calculated increase of G-74 that was added to the PG Stream.

With valve 147 torque-closed and either 57 or 61 open, allow the PG stream coming from the "Incoming Header" to flow down to the Tube Rack and on past to the "Outgoing Header", by passing through the Venturi Meter and Gas Mixer. Now with no flow of dry air going through the rotameters and thus no flow through valve 57 or 61, take the following readings:

With Q = 0 scc/min

M (DBM) ________	A S_{28} ________
G (PBM) ________	B S_{20} ________
T_v (Venturi Temp.) ________	C S_{14} ________

[redacted]

T_r (Rotameter Temp.) ______________ E S_{32} ______________

Pirani
Q (Flow) ______________ F S_{44} ______________

After these readings have been made, open valve 101 or 102 until an approximate flow of 100 cc/min is recording on the rotameter. Repeat the readings as shown above and calculate the change in S_{14}, by using the following equation:

$$\text{Factor "F"} = \frac{S_{14} \quad \text{(of 100\% Air Sample)}}{1 + \frac{63}{Q}\sqrt{\frac{T_r\ m\ (G = 063m)}{Tv\ p\ (1 - .00919\%\ x\ .0091\%O_2}}}$$

Where:

Q = Rotameter Reading in Scc/min

T_r Rotameter Temperature in degrees Rankin **

T_v Venturi Temperature in degrees Rankin

M DBM Reading

G PBM Reading

P Pressure in Manifold Box (psia)

$\%N_2$ % Nitrogen in Stream

$\%O_2$ % Oxygen in Stream

If the factor "F" obtained from the formula above varies more than 2% from the original value, the machine should be recalibrated.

The use of the calibration check is only tentative and no proof of its accuracy has been made. As soon as experience shows this check to be satisfactory, more information will be issued.

[redacted]

** Degrees Fahrenheit + 460.

C. Residual Checks of the L.R.

The residual check of a Line Recorder consists of pumping out the process gas on both sides of the adjustable leak for either the "A" or "B" machines until T.C. 2 of the manifold pump reads 10 microns or less.

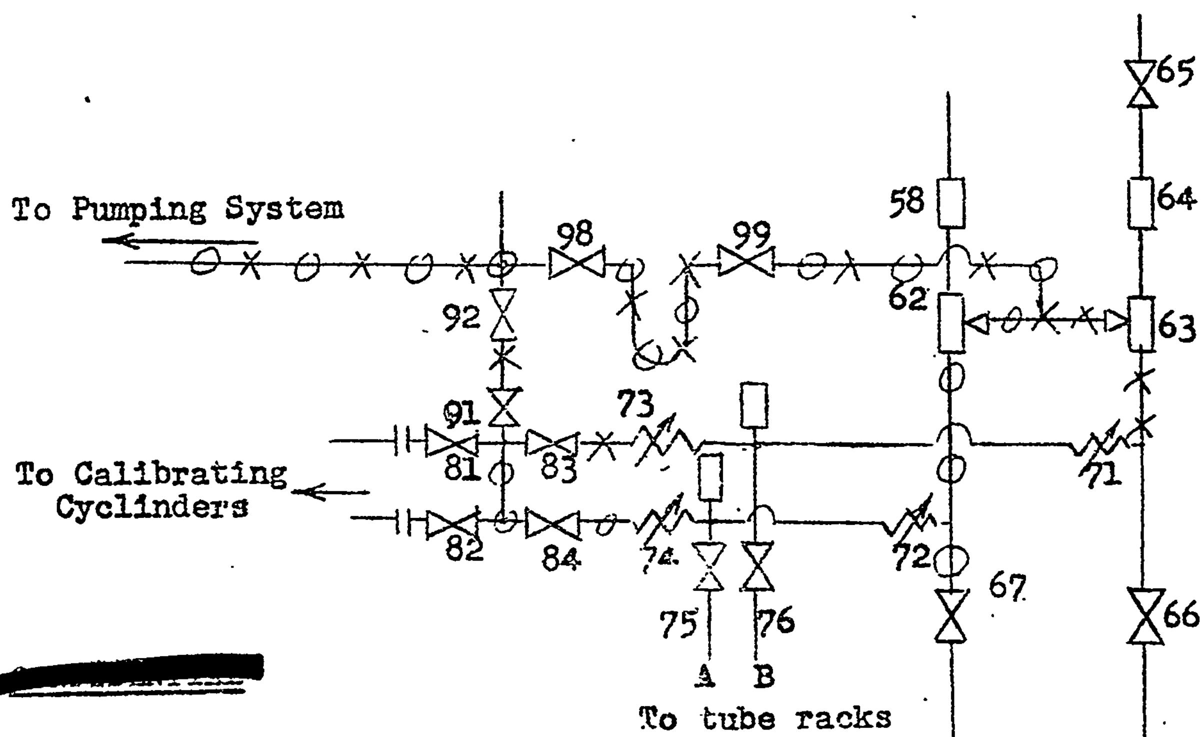
The Procedure Is:

1 - For Upper Loop - Torque valves 59, 67 & 58 to 30 foot pounds.

For Lower Loop - Torque valves 64, 65 & 66 to 30 foot pounds.

2 - Close the calibration and Process adjustable leaks.

3.- Pump behind the calibration leak and behind the process leak as shown below (Lines indicated thus: OOOOOOO & XXXXXXX)



[redacted]

4 - Adjust bridge current on the Pirani gage to the value given on the calibration chart.

5 - When mass 28 is constant for three readings, take the readings as listed on the succeeding page and record.

6 - <u>If the Pirani gage does not read [illegible], consult your shift supervisor, but do not make adjustments on this gage.</u>

D. <u>100% Air Checks on the L. R.</u>

The 100% air check for determining the accuracy of calibration for the L. R. is conducted immediately after making the <u>residual check</u>. This procedure is:

1 - Open valves 92, 91, ~~and 83 or 84~~ in the order given.

2 - Close valves 62 or 63, 99, 98, ~~and~~ 96, and 83 or 84 in the order given.

3 - Open valve 100 to admit a sample of air into the lines.

4 - Close 92 and 91.

5 - Open valve 83 or 84 and adjust the Static Calibration leak until the Pirani Gage is recording the correct value. (Refer to the Calibration Chart).

6 - Allow enough time for the readings to become constant and tabulate them at the bottom of the Residual Table

<u>The last line of this table is to be filled in by the shift supervisor.</u>

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LINE RECORDER RESIDUALS

BUILDING:________ LINE RECORDER________ DATE:________ STARTED PUMPING AT:______

READINGS TAKEN AT: ______

AMP. ZERO	PIRANI BEFORE ADJUST.	I.G. X 10^{-6}	A READING	B READING	C READING	E READING	F READING	H READING
			SENS.	SENS.	SENS.	SENS.	SENS.	SENS.
			CONSTANT	CONSTANT	CONSTANT	CONSTANT	CONSTANT	CONSTANT
			RESIDUAL %	RESIDUAL %	RESIDUAL %	RESIDUAL %	RESIDUAL %	RESIDUAL %

PROCEDURE: UPPER LOOP Torque valves 59, 67, and 58 to 30 ft. lbs.

LOWER LOOP, Torque valves 64, 65, and 66 to 30 ft. lbs.

Pump behind the calibration leak and the process leak.
Adjust bridge current on Pirani to the value given on the calibration chart.
When the mass 28 peak is constant for three readings, tabulate.
DO NOT CORRECT FOR AMPLIFIER ZERO.
Taking the temperature correction into account, adjust the Pirani.

Make a ~~100% air~~ check and record below.

Amp Zero_____ Pirani_____ C:____X____X____ = _____%G-74 E:____X____X_____ = %O_2

I. 95% Check

For an efficient check of a Line Recorder using approximately normal contaminant percentages the 95% - 0.61%, 5% A-74 gas check is employed.

This check is quite similar to the 100% [illegible] check except that the gas admitted is obtained from a known-mixture gas sample.

The procedure is:

1 - For upper loop - Torque valves 69, 67 and 63 to 30 foot pounds. For lower loop - Torque valves [illegible], 65 and 66 to 30 foot pounds.

2 - Close the calibration and process adjustable leaks.

3 - Pump behind the calibration and process leaks by: a. Opening 96, 98, 99, 62 or 63, 92, 91, 82, 84 & 80 or 81, 83 & 79.

b. Leave these valves open until the residual readings of the machine are normal (see taking of Residuals Page 114).

4 - Close valves ~~92 and 91~~. 62 or 63, 99, 98, 97, and 91.

5 - If the gas sample is in the lower bottle, crack 78 for a very short period of time. If the sample is in the upper bottle crack 77.

6 - Close 78 & 77.

7 - Open 84 or 83 and adjust the calibration leak until the Pirani reads 50 (Corrections may be necessary)

[redacted]

If the pirani is too low repeat procedure [illegible] sensitivity controls on [illegible] amplifier [illegible] if necessary.

8 - When mass 28 is constant for three readings, take the readings of the A, C, and E channels. If the readings correspond in percentage to the known sample composition,** the machine is "on calibration". If the oxygen or E channel reading is more than 5% above the residual reading, the entire 95% - 5% check should be re-run.

9 - When the check has been completed, close valves 80, 82, or ~~29~~ 78, 81, ~~83 or 85, 33, 38~~ and the calibration leaks. Open 92 and 91 and pump out until the pirani is zero. ~~Then close 83 or 84, 91 and 92.~~

10 - Open valves 59, 67 or 55 and 66.

11 - Put the machine on stream by opening the adjustable leak until the correct Pirani reading is obtained.

12 - Adjust sensitivity controls to proper readings.

If this check proves that the machine is out of calibration, notify your supervisor.

NOTE: Erroneous readings for masse 20, 32, 44 and 69 prove that the gas sample has been contaminated. The 95% - 5% check is then inaccurate.

** These readings are to be within 2% of the known sample composition.

[redacted]

Pumping Behind The Calibration Leak

The valves which block the flow of contaminant gases to the calibration leaks of the [illegible] have been found to be faulty and due to this, inleakages of contaminant gases into the Tube Racks are present.

To alleviate this trouble the following procedure should be used at the beginning of each shift.

1 - If the machine is operating normally close valves 73 or 74.

2 - Open valves 92, 91 and 83 or 84 in the order given.

3 - Allow valves 83 or 84 to remain open until the pressure as measured with TC [illegible] is 10 microns or less.

4 - Close 83 or 84 but continue pumping on the line throughout the shift.

It will be seen that pumping on valve 83 or 84 and the Calibration Leak continuously is not practical for several reasons. However, this practice should be employed as much of the time as possible.

[redacted]

4. <u>Sampling Procedure</u>

1. <u>Equipment</u>

During normal operation, the Hoke tube sampling manifold is continuously being evacuated between valves 90-1 and 86. (See Fig 3)

The sample tube is joined to the manifold by a special union at 120-3 and is evacuated by opening valves 86 and 85. It is advisable to have the sample tube opened and to evacuate it together with the entire system.

A nickel U-tube is soldered to the Sampling Manifold between valves 90-1 and 89. Valve 88 is connected to a thermocouple gage which is a measure of pressure in the manifold; valve 87 is connected to the evacuation header and pump. Valves 86 and 90 are straight-through valves permitting gas flow when in the open position.

Valve 89, connected to a G-74 line for use during purging, is kept closed during the entire sampling procedure. Since it is a tee valve, flow is straight through when in the closed position.

The sampling manifold is electrically traced and is heated to a constant temperature of 106° C. The U-tube is equipped with Calrod heaters and a Thermal Element with which the enclosed solid sample can be heated to 106° C, and is vaporized at the time of transfer.

[redacted]

2. Sampling Operations

1 - If the cell from which the sample is scheduled to be taken is not being analyzed with either tube rack, the Station operator should make necessary change as described on Page 101. It is desirable to make this change on the loop connected to the "spare recorder" if possible.

2 - The Hoke tube should be in place with the union tight and evacuation of the sampling manifold up to valve 90-1 should be maintained until the Thermocouple gage indicates 20 microns or lower. Occasionally this degree of vacuum is not readily obtained even after the valves are tightened. In this case, it is permissable to start sampling if the pressure is below 75 microns.

3 - Place a cold trap containing a slush mixture of dry ice and trichlorethylene under the nickel "U Tube" of the Hoke tube sampling manifold. Allow 5 minutes before starting to draw sample.

4 - Open valve 68 if sampling from the upper loop or valve 69 for the lower loop. Close valve 88 and 86 but keep valve 87 open.

5 - Open 90-1 for a period of 15 seconds to obtain the desired weight of sample. This time is subject to slight change depending upon the weight of sample desired. Experience will aid in determining the proper sampling time.

6 - Close 90-1 and then close and torque valve

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68 (or 69). Open 90-1 for a few seconds to pump out the line up to 68 (to prepare for the next sample) and then close 90-1 again.

7 - Continue evacuating through valve 87 (with the slush trap still cooling the "U" Tube) for 2 or 3 more minutes.

8 - Close valve 87 (88 should still be closed) and place the slush trap under the Hoke sample tube covering only a one-inch section.

9 - Turn "U" Tube Switch on panel to "on" position so that the Calrod heaters may heat the "U" Tube to drive the C-616 sample into the Hoke tube. While this is heating (it may take 5 -20 minutes) fill out the sample cards as described on page______.

10 - When the "U" Tube goes off, the "U" Tube has reached the proper temperature and the switch can be turned off. Shut valves 85 and 86 tightly and open valve 87 to start pumping down. After two minutes, valve 88 to the Thermocouple gage may be opened.

11 - Disconnect the union 120-3 and attach the sample cards to the Hoke tube. Place a new tube in position for the next sample and again pump this system down.

12 - The information required in the Sampling Log Book should be entered.

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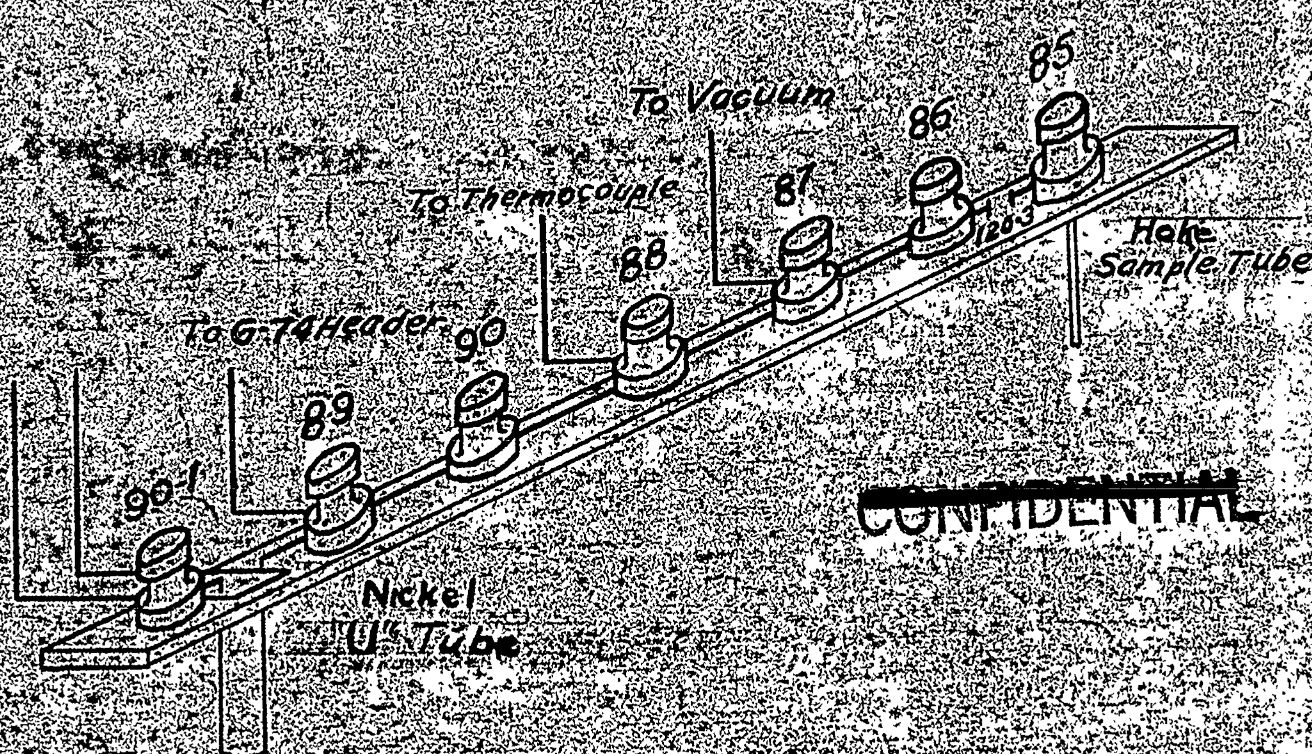
FIG 3



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SAMPLING EQUIPMENT

FILLING OUT OF ASSAY SAMPLE CARD

The samples withdrawn in either Hoke tubes or "U" tubes must have identification tags fastened to them before the samples are sent to the laboratory for analysis. These tags or cards must contain the following information:

1 - The building number that the sample was withdrawn from.

2 - Cell number that the sample was withdrawn from.

3 - Date and time that the sample was withdrawn.

4 - Number of sample.

5 - Destination of sample - Laboratory "B" or "C". (This is accomplished by circling the correct letter.)

6 - Distinction of Hoke Tube or "U" Tube.

7 - One card for a receipt to show that the sample has been picked up by the messenger.

Once the sample has been turned over to the messenger, he will sign the "blue" copy and leave it in the station. These cards are to be filed for future reference. Also the operator that has drawn the sample must sign his or her name to the back of the third copy (blue card).

The correct method to fill a set of sample cards out is illustrated on the following page.

[redacted]

122

~~CONFIDENTIAL~~

MODEL SAMPLE CARDS

302-5 No. 150 B C

1 2 3

Sample Requisition

Cell 6

DATE

11:00 A. M.

6/5/45

L. R. 302-5-67*

Remarks:

White Card

If "U" tube sample is taken, just write "U" tube in the space.

302-5 No. 150

DO NOT FILL OUT.

Rec'd By

Pink Card

302-5 No. 150 B C

Cell 6

302-5-67*

11:00 A.M.

6/5/45

Messenger

Blue Card

B. Tube Rack Operations

1. GENERAL:

The operation of the tube rack is continuous and automatic as long as proper operating conditions are maintained. The operator should see that the cold traps are kept filled (see discussion on page 178), the proper pressures are maintained with the diffusion pump and the fore pump, and that the proper sample flow rate into the M.S. tube from the Manifold is maintained.

Because of the special construction of the tube rack, care should be exercised to avoid the following:

1 - Jarring the T. R. equipment because the proper alignment of magnets, etc., must be maintained.

2 - Touching electrical connections carelessly, since some are maintained at relatively high voltages.

3 - Placing objects on the T. R. supports or in other positions where they might damage the fragile equipment if they were to fall.

4 - Spilling slush on mercury bulb of chemical trap which prevents the mercury from vaporizing properly.

5 - Bumping glassware so as to cause de-gassing and loss of vacuum.

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2. Thermocouple Gages and Operation

A thermocouple gage is a device used to measure low pressures in the range of 200 down to a few microns. Current from a battery is used to heat a wire in the T. C. gage tube and a sensitive thermocouple is provided to indicate changes in the temperature of the wire. The current from the thermocouple rises with increasing temperature and is read on a microammeter which is calibrated to read microns directly. If the pressure in the T.C. gage tube rises, more heat is conducted away from the heated wire and the reduced thermocouple current results in a higher pressure reading on the microammeter.

It is important that the current through the heated wire be kept constant at the value set during the calibration of that Thermocouple gage. Therefore, a variable resistance and a milliammeter have been provided to keep the desired current as the battery runs down. When the dial indicates that the battery should be replaced, maintenance should be notified.

Thermocouple gages are provided on the tube rack (1) at the discharge of the diffusion pump and (2) at the suction of the fore pump. Two gages have been provided so that these pressures can be measured if the diffusion pump is isolated from the tem. This might occur if the fore pump were being used to pump down the gas lead before opening it to the M. S. tube. (See page______)

The two thermocouple gages are connected, through a switch, to the same milliammeter circuit and microammeter. Because of slight differences in the circuit resistances, the heating current must be different for each thermocouple gage. To read T.C. gage #1, turn switch to position "1", adjust resistance until the milliammeter reads the current for #1 as labelled, then read the pressure from microammeter. Similar procedure is followed for T.C. gage #2. When not in use, turn switch to "off" position.

The Thermocouple gages on the manifold are identical in construction and operation. (See page 304).

3. Diffusion Pump Operation

The diffusion pump provides a means of attaining the high vacuum (10^{-7} - 10^{-5} mm mercury) essential to the proper operation of the M. S. tube. Molecules from the space being evacuated diffuse into a stream of high-speed mercury molecules which push the former molecules to the diffusion pump discharge from which they are drawn into the fore pump. The mercury is condensed and re-used by vaporizing it by means of an electric heater.

The mercury vapor is forced through a small opening so that the high-speed jet is produced.

Proper operation of the diffusion pump consists merely of maintaining three conditions.

1 - A low diffusion pump discharge pressure must be maintained by the fore pump. If the mechanical pump fails, the diffusion pump ceases to function after a period of time.

2 - The heater must be on to vaporize the mercury. A light on the tube rack panel indicates that current is supplied to this heater.

3 - Cooling water must be circulated through the water jacket to condense the mercury vapor. The pipe leading to the drain should be checked often for proper flow rate. Failure of the water supply is discussed on page 303.

4. Fore Pump Operation

The fore pump (also called a mechanical pump, rough pump or Welch pump) is used to keep the diffusion pump discharge at a low pressure or for miscellaneous low-vacuum evacuation.

The usual construction of these pumps consists of an off-center rotor with movable fins so arranged that as a slug of gas is sucked into the cylinder of the pump, the previous slug of gas is forced into the oil-immersed outlet port and to the atmosphere.

As long as this pump has the proper amount of oil maintained in it, and no water, mercury, C-616 or C-816 admitted, it will pump down satisfactorily to a pressure of 10 microns (.01 mm mercury) or less. Continuous operation under the above conditions may be expected, and in fact the longer the pump operates, the better it becomes.

5. <u>Filling Cold Traps</u>

Cold traps are necessary to condense certain vapors whose entry into a system might prove harmful to that system. The coolant material is either liquid nitrogen (L-28) or a slush mixture of powdered dry ice (CO_2) and trichlorethylene. The L-28 vaporizes at -197°C while the temperature of the S-44 slush is approximately -78°C.

Each tube rack has an L-28 cold trap between the M. S. tube and the diffusion pump. This serves the dual purpose of preventing back-diffusion of mercury vapors from the diffusion pump into the M.S. tube and any condensable vapors (such as C-616, C-816, etc.) of the M. S. tube from entering the diffusion pump. (Normally, a negligible amount of these vapors are condensed in this trap since the chemical trap removes the C-616 and the cooling water condenses the mercury).

The L-28 cold trap should be filled once per shift or more often if found necessary.

Although the cold-trap crew usually attends to this duty, it is the <u>responsibility of the operator</u> to maintain normal operating conditions by checking all cold traps frequently.

The criterion for refilling L-28 cold traps, is that at least 2 inches of L-28 be maintained in the trap. <u>It is not necessary to fill these traps every two to three hours</u> - once a shift should be sufficient, however, the traps are provided with a low-level alarm that sounds in case the L-28 level becomes so low that the trap temperature exceeds a certain set point.

The L-28 cold trap is filled using a metal can with a spout for transferring the coolant to the Dewar flask. Safety goggles and gloves shall be used while transferring the L-28 from the carboy to the cold trap.

The chemical trap requires a cold trap filled with S-44 slush for proper operation. These slush mixtures are prepared by filling the Dewar flask with powdered dry ice and adding just enough trichlorethylene to make a <u>thick</u> slush. Stirring should be done with a <u>wooden</u> rod, and great care should be exercised during stirring and usage to keep from breaking the fragile flask. (In re-filling the flask, it has been found that enough trichlorethylene is usually present so that only dry ice need be added before stirring.

The slush trap serves to maintain a "cold point" where the reacted gas of C-616 and mercury vapor condenses. (See page 9). The heat is conducted from the collar surrounding the cold point through a copper rod into the cold trap. It is essential to keep this system cold; otherwise mercury and C-616 will enter and damage the I. S. tube.

Water ice collects on the copper rod by condensation from the air. The slush level should not be above the 5/8-inch copper rod or too much ice forms. It is necessary that the operator scrape off the ice formed on the <u>collar and PG inlet tube</u> to keep the cold point from shifting to the wrong location.

Covers should be kept on the flasks to reduce the amount of water getting into slush mixtures. A schedule will be set up whereby the slush traps are replaced weekly so that the water collected can be removed.

The L-28 cold traps on the evacuation system of the manifolds must receive the proper attention as described above for the tube rack cold traps. Instead of once per shift, these cold traps should be checked every 2 to 4 hours. Checking these traps is another responsibility of the Station Operator,

[redacted]

[illegible] to [illegible] that [illegible]

[illegible] form in the [illegible] Dewar flask.
If this ice does form, gently push it away
from the glassware, making sure that no ice
falls into the flask. Periodically, these traps
are to be emptied and all ice removed. Also,
the large "U" trap which is cooled with water,
should be filled [illegible] the water-ice content
[illegible] Further instructions
regarding this trap will be found on page 153

Safety precautions must be exercised
to keep from getting "burned" by the extremely
cold materials used. Goggles and gloves are urged
by the Safety Department. (See also pages 147
and 148).

~~CONFIDENTIAL~~

C. Relay Rack Operation

In the description of the Relay Rack, (part I-D-3 of this manual) the various panels and their functions were described. As explained previously, these panels require periodic adjustments and checks in order that the Line Recorder may operate satisfactorily. The adjustments and checks necessary on the L. R. equipment, once it has been calibrated and turned over to the L.R. Department, are as listed below:

1. Ion Gage

This electronic circuit is employed to measure pressures within the M.S. tube below a value of 5×10^{-5} mm of Hg.

Periodically, twice or more per shift, the operator will check the Ion Gage as follows:

a. Zeroing the meter

1- The Scale Factor control is turned to the "off" position.

2- The Instrument control shall be set to "Ion Gage Automatic".

3- If the meter does not read zero, turn the Balance Control until the meter is reading zero.

b. Checking Sensitivity

1- Turn the Instrument control to "Check Sensitivity".

[redacted]

2. The meter should read "5", and if not, adjust the <u>Sensitivity</u> knob until the meter is on "5".

c. <u>Checking Emission</u>

1- Turn the <u>Instrument</u> control to "Check Emission" and adjust the <u>Regulate</u> knob until the emission plateau is reached. (Contact your supervisor if this procedure is necessary).

2- Turn the <u>Emission Adjust</u> control until the reading is "8" - if necessary.

3- The sensitivity must now be re-checked and if the controls are changed, the Emission and Sensitivity are checked against each other until Sensitivity reads "5" and Emission reads "8".

d. <u>Normal Operation</u>

1- The Ion Gage shall be operated at all times on the <u>Instrument</u> setting of "Ion Gage Automatic". This then provides the safety cut-out feature, in case of high pressures, for the two filaments.

2- For normal operation the <u>Scale Factor</u> control will be set to either "1×10^{-6}" or "1×10^{-7}" readings. Pressures above these two settings should be brought to the supervisor's attention immediately.

e. <u>Special Operations</u>

If at any time the pressures cause the gage to cut-out the procedure is:

1- Allow a reasonable length of time to elapse so that the tube pressure may be pumped down.

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CONFIDENTIAL

2- Push the Filament On switch with the Instrument switch on "Ion Gage Automatic" and the M.S. tube Filament switch to "off".

3- If the Ion Gage remains on the pressure is below 1 x 10^{-6}, the Filament switch on the Emission Regulator may be turned "on" and normal operation be continued.

4- If for any reason the pressure is not pumped down within 15 minutes after the disturbance, check with supervisor.

At times the Ion gage must be turned off. To do this, push the Filament Trip button. When turning on the filament of the Ion gage follow the procedure as described above.

"Degassing" of the Ion gage tube is at times necessary due to the high pressure that results from the slow degassing of absorbed gas under normal conditions. The procedure is:

a. Throw Filament switch of Emission Regulator is "off".

b. "Trip" the Ion gage filament.

c. Turn the Grid switch on for a period of 1 minute.

d. Put the Filament control of the Ion gage to the "on" position.

e. Now throw the Grid switch to "on" alternately every 30 seconds until the pressure as read on the Ion gage meter does not increase more than 2 large divisions with a scale-factor of "1 x 10-6". When this condition is obtained, return to normal operation settings.

[redacted]

2. Emission Regulator

The design of the L. [illegible] equipment is such that the number of ions for a certain percentage of contaminant must be kept constant, if accurate results are to be obtained. This is accomplished by holding the number of electrons emitted from the M.S. tube filament to a certain constant value with an emission regulator.

To check this Emission Regulator the procedure is:

a. Turn the Filament switch to the "on" position.

b. Adjust the Regulate knob until the emission plateau is reached.

c. Adjust the current of the filament with the Emission control knobs so that the emission is reading the value designated on the Calibration Chart.

d. Check the reading for the Trap current. If this value is not within 80% of the Emission current, notify Maintenance.

To read correctly the Emission value, the reading of the Emission Meter is to be multiplied by the sensitivity factor at which the instrument control is set.

3. D. C. Amplifier

To assure correct amplification of the signals from the M.S. tube pre-amplifier and amplifier, certain conditions must be maintained. These include maintaining the correct filament emission of the amplifier tubes, proper voltage regulation, proper battery voltage,

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and correct amplifier zero setting.

These conditions may be checked as follows:

a. <u>Filament, Regulate and Battery Checks</u>.

1- Turn the <u>Check</u> switch to "Fil." and then to "Reg.". The meter should read within the "Red Band" range for both and also when the "Bat." switch is depressed.

2- If the meter does not read within this range for the three checks, the shift supervisor may correct the condition by adjusting the proper screwdriver controls in the back of the panel.

b. <u>Amplifier Zero</u>

1- The Amplifier Zero is corrected by putting the sensitivity control of the Output meter to factor "x 1", while the L.R. is on a point which requires no acceleration voltage. (Pirani or Amplifier Zero prints).

2- Adjust the "Fine" balance control to make the necessary correction so that the meter reads "O" and immediately turn the output meter to "off" before the machine starts reading a peak which requires acceleration voltage. This adjustment is made <u>only</u> when the print point is 1 or more divisions either side of the zero line on the recorder strip chart.

3- If the "Fine" balance control is not sufficient to balance the meter to zero the procedure is:

CONFIDENTIAL

a. For too low meter reading, turn the "Fine" control counter-clockwise to the low position.

b. Revolve the "Medium" control clockwise One position.

c. Adjust the "Fine" control for the proper meter reading.

If this does not bring the reading to zero, repeat the procedure until the correct reading is obtained.

a. For too high a meter reading turn the "Fine" control clockwise to the far position.

b. Revolve the "Medium" control ONE position counter-clockwise.

c. Adjust the "Fine" control for proper readings or repeat the above procedure, if necessary.

c. Sensitivity controls, for the 8 channel signals printed by the Micromax recorder, are provided so that the Micromax will not print off-scale for any mass. These controls are adjusted for an increase in reading by turning clockwise one position at a time, and for a decrease in reading, counter-clockwise.

The sensitivity settings are x100, x50, x20, x10, x5, x2, and x1 for the recorder while for the Output meter the sensitivity settings range from x1000 to x1. The reason for this difference in sensitivity controls is that the Output meter is ten times more sensitive, hence for the same reading on the recorder and the Output meter the sensitivity setting for the Output meter will be 10 times greater than for the recorder.

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in some cases (such as for mass 28), the lowest sensitivity is too high for the amplifier output above certain concentrations of that component. In these cases it is advisable to turn the sensitivity to "Off".

4. <u>Main Control</u>

This panel houses the necessary screwdriver controls for the Micromax Recorder, the Focus and Trimmer adjustments, Acceleration Voltage Selector & Point Selector switches and the Manual Divider.

a. The Focus control setting is determined by the static calibration and is to remain constant at the value specified on the Calibration.

b. The Trimmer control settings are specified on the Calibration Chart, but as the machine is used these settings are changed due to "drifting" of the High Voltage Supply which necessitates repeaking of the machine once per shift.

c. <u>Peaking-Trimmer</u>

1- With the Line Recorder operating correctly, and printing mass 28, turn the output meter control of the D.C. Amplifier to the sensitivity that will allow the meter to read approximately half-scale.

2- Using the Trimmer "Fine" control, adjust until the meter reads the highest value(maximum). Only a slight adjustment is usually necessary.

d. <u>Peaking - Trimmer and Screwdriver</u>

1- Follow the two steps given above for

[redacted]

rimmer peaking.

2- Turn the Output Meter control to the sensitivity that allows the meter to read approximately half-scale for each signal as measured.

3- Adjust the screwdriver control for that mass (Channels must correspond) so that the Output meter reads the maximum signal

4- Follow the last two steps, (2 & 3) of this procedure for all masses.

e. Precautions

1- Peaking must be accomplished quickly, for a period of only 24 seconds is needed for the machine to print and then change to another mass.

2- Always turn the Output meter control to "Off" a few seconds before the signal is printed. If this is not done, damage to the Output meter may result.

d. Once a week (or more often if necessary) the machine constant (acceleration voltage x mass) shall be calculated for each measured component. This is done by multiplying the acceleration voltage for each component measured, as read on the voltmeter, by the weight of the mass for that component. If the machine constant, for each component gas measured, equals the average of the readings within 5%, it can be assumed

[redacted]

Continuedd from Page 138

all masses.

2. Record the Trimmer Fine Setting for each point printed. (Neglecting Pirani & Amp. Zero)
3. Add all the Trimmer Fine settings and divide by the number of settings, thus determing thé average.
4. Enter the Trimmer Fine Setting for each point and Average Setting on the Data Sheet Provided.
5. Set the Trimmer Fine control to the Average Setting reading.
6. Notify your supervisor immediately if any readings differ more than "2" divisions from the average setting. He will then peak the machine with the screwdriver adjustments and Trimmer control.

E. Precautions.

1. Peaking must be accomplished quickly, for a period of only 24 seconds is needed for the machine to print and then change to another mass.
2. Always turn the Output Meter control to "Off" a few seconds before the signal is printed. If this is not done, damage to the Output meter may result.

F. Summary

If the Trimmer Fine setting for the channels are within 2 divisions of the average setting, it may be assumed that the Line Recorder is peaked correctly for all masses (components). If however, some of the readings seem erroneous, further checks should definitely be made.

5. High Voltage Supply

This unit supplies the voltage necessary for the M.S. tube accelerating plates. The visual check for determining if the H.V. supply is operating properly is the reading of the milliammeter. This reading should be within the "Operating Range" of 0.2 and 1.2 m.a. Otherwise, contact your supervisor.

6. Micromax Recorder

Once the recorder is operating properly, it is automatic and only a few mechanical aspects of this instrument need be checked by the operator. These are:

1- Inspect the Strip Chart to determine if it is properly installed. If the chart is near the end of the roll (30 in.), contact Maintenance.

2- If the print points are not clear, see that the ink pads are re-inked or replaced by Maintenance.

3- If the Amplifier Zero reading is not within 1/3 division of the value indicated by the Output meter, or the Pirani reading is not within 1/2 division of that indicated by the Pirani gage, notify Maintenance.

[redacted]

4. To change C.C.R. from one L.R. unit to the other, open the door behind the panel in which the "principal" recorder is located. Unscrew and remove the <u>CR-1 Receiver</u> plug and install this line in the <u>CR-1 Receiver</u> behind the Recorder of the other L.R. unit. Be sure to notify C.C.R. of this change.

5. If at any time it is necessary to revise the printing program of the L.R.'s, the new program plugs will be installed either by Maintenance or by the L. R. supervisory personnel.

7. <u>Main Switch Panel</u>

The precaution to be observed with this panel is to check periodically to see if any switches have accidentally been thrown to the "Off" position. Only in case of an emergency, where the personnel or equipment is endangered, should these switches be thrown without specific instructions from the supervisors.

[redacted]

[illegible]

1. Strip Chart

The following information will be recorded on the strip chart:

a. The time, date and operators initials will be entered at the beginning of the shift and the time will be recorded hourly thereafter or whenever a major entry is made.

b. At the beginning of each shift, and whenever there is a change, the program number and all sensitivities will be recorded. When the sensitivities are changed, the channel, the new sensitivity and the time will be tabulated.

c. Anytime that a change is made which is not due to a change in process (e.g., adjusting leaks, adjusting Amplifier Zero, filament off, etc.), it should be indicated on the chart.

d. Any pertinent data relating to Process should also be indicated.

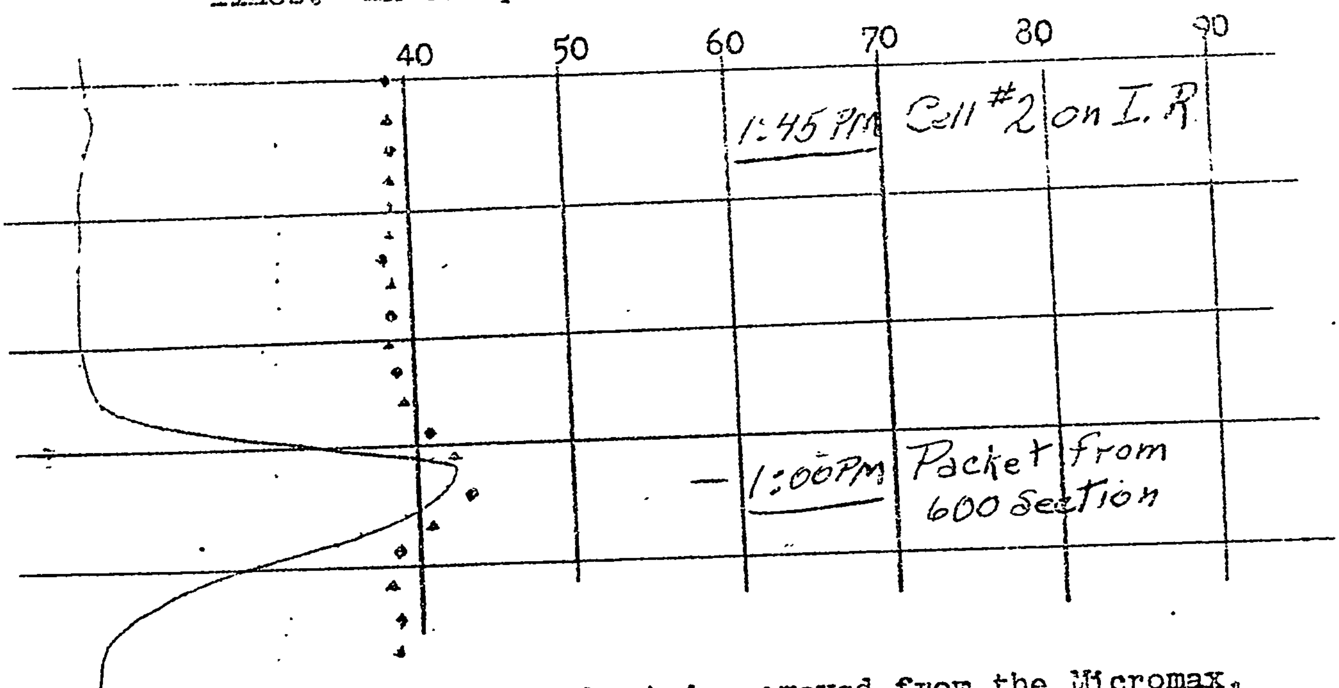
The method used to obtain the correct readings are:

a. The hourly readings for entry in the Log Sheet shall be based on the average of the readings for the past 10 minute period, unless there is a packet, surge or Pirani fluctuation.

b. In such cases the readings may be taken when they become constant-not to exceed 20 minutes after the hour, or if necessary 10 minutes before the hour.

[redacted]

All entries on the strip chart shall be neatly printed between the "60" and "90" division lines. An example is:



When a chart is removed from the Micromax, it is to be filed in the main office. *operate correctly, under any conditions*
Could any of your I.R. Units
with the Pirani reading as shown? Refer to page 102.

NOTE: WHENEVER A CHANGE IS TO BE MADE ON THE LINE RECORDER ALWAYS NOTIFY THE CCR BEFORE CONTINUING WITH THE CHANGE.

The Station Operator should remember that the condition of the strip chart, log book, and log sheets is an important indication of the condition of that Station. Therefore, make all entries neat, accurate and complete.

[redacted]

2. <u>Log Sheets</u>

The log sheets are made out in order to give a general picture of process conditions for a particular building. Each hour, all data called for on the log sheet will be recorded. "Remarks" is to explain any readings on the sheet which would tend to give false impressions of the condition of the process condition.

If a tube rack is being repaired or standing by, operators should continue to record ion gage, thermo-couple gage, etc. so that the equipment will not "go bad" during the shift without the operator's knowledge.

3. <u>Log Book</u>

The log book should supplement the strip chart and log sheet. It should contain all pertinent information regarding the process of L.R., all non-routine actions, suggestions, a complete record of the process stream as obtained from Building operations, comments of any kind, instructions that should be forwarded to the next shift, etc. All entries should be initialed by the operator.

The data obtained from each cell scanned will be entered in the data sheet provided for that Line Recorder. Also note to which cell you have changed the analyzing point.

When it is necessary to take data for entry in the data sheet and the operator's time is being consumed on some other job, it is only necessary that he mark the correct time on the strip chart so that when time allows, the data may be read off the chart and entered on the sheet. However, Readings that are not printed by the

 [redacted]

Micromax, must be recorded at the proper time.

4. Miscellaneous

Other information which should be recorded by the operator is as follows:

1 - Peaking Data -

This data, which consists of the Coarse and Fine Trimmer control settings, the current and voltage readings of the High Voltage supply and the initials of the operator doing the peaking, shall be kept on the "History of Peaking" log sheet that is posted on the panel board.

The operator will peak the machine once per shift with the trimmer, while the machine is recording Mass 28. The supervisor will peak the machine with the Trimmer and Screwdriver adjustment on the 3rd shift of the 7th day and note the deviation of reading for: Mass 14, 28 and 32. At the end of the work week (Sunday -3rd shift) the sheets will be collected and sent to the C.C.R. There the results of the peaking will be analyzed and the information used.

2 - Residual Checks -

These Residual sheets provide spaces for the following information:

Building, Line Recorder, Date, Time the Pumping was started, time of Readings and the Readings - Amplifier Zero, Pirani setting, Ion gage pressure, reading, sensitivity and constants of all masses and the Residual Percentage of all masses (Signal

[redacted]

X Sensitivity X Calibration Constant).

Also, at the bottom of the sheet there is space for entry of the 100% air check results.

When the sheet is filled out the shift supervisor will check and send it to the area supervisor for reference.

3 - Top & Bottom Cell Scans -

Once per shift the top and bottom cells of each building (usually #1 and #2 cells) are analyzed to check the G-74, O_2, CO_2 and C-816 concentrations.

The results of these scans shall be tabulated in the log book and also telephoned to C.C.R. for their files. The results will consist of:

Time, Date, Building, Cells analyzed, Line Recorder used for the check and the actual percentage readings for the two cells.

4 - Status of Building and Station -

The condition of each building and station and the number of assay samples withdrawn is to be reported to the area supervisor at the end of each shift. This report is to enable the next area supervisor to more quickly decide what will be done on his shift.

[redacted]

TO: All L. R. Department Supervisors and Operators

FROM: J. C. R. and L. R. - Process Coordinators

SUBJECT: Instructions for forwarding scanning information to the L. R. - Process Coordinator.

1. A "T and B" (top and bottom cell) check is requested for each building as early as possible on the 4 - 12 shift each day

   (a) J. C. R. should be called before scanning to determine if conditions are stable enough to permit obtaining reliable results.

   (b) Use the phone number 25 when calling for this information and contact the L. R. - Process Coordinator.

   (c) All scanning data is to be entered on the form of the kind attached to these instructions. Operators will fill in the blanks that are underlined. Constants for any machine need be entered for one set of readings only

   (d) The completed forms will be picked up by the coordinator or his assistant. Notify C. C. R that these forms are complete, as they are completed.

2. Accurate readings are of utmost importance.

   (a) Sensitivity settings should be for highest value permissible (lowest factor) such as C x 1 instead of C x 5

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146-A

(b) Readings should represent the average values for a period of 30 minutes.

(c) The L. R. strip chart should be marked with an arrow to indicate the point at which readings were taken.

(d) T and B Readings should be taken with the pirani setting as near as possible to correct setting at this concentration. This should result in pirani readings the same or within 2 divisions in both cases.

3. Building Foremen and crew leaders have been instructed by the general foremen to advise the station operators on the following information for their building.

(a) Stage pressures for each building.

(b) Condition of each cell in building.

(c) Changes in cell status in advance.

This information should be indicated by:

(a) Giving the time and date of last air or 95-5 check.

(b) Giving time and date of last calibration.

NOTE: This procedure supercedes #3 of Page 146 (top and bottom cell scans).

[redacted]

I. Miscellaneous:

1. <u>Safety</u>

The general subject of safety is vitally important both to the individual and to the whole organization. Through carelessness, the individual stands to lose both time and pay while the organization loses the services which are badly needed at all times.

Carbide is very Safety-conscious as evidenced by the active Safety Department. Although operation of the Line Recorder Station is by no means a "dangerous job", nevertheless precautions should be taken to avoid harm. Some of these are:

1 - Keep equipment in the proper place. Don't leave sampling blocks or torque wrenches where they can be tripped over.

2 - Use care in handling L-28 and dry ice. Treat these with the same respect as you would boiling water---both refrigerants are cold enough to cause bad "burns". Gloves and Safety goggles are issued to every operator from the Safety Storeroom in 309-3.

3 - Refrain from breathing trichlorethylene fumes. These can be harmful if inhaled in sufficient quantity.

4 - DRY ICE IS <u>NOT</u> TO BE USED TO COOL MILK OR SOFT DRINKS. There is danger of injury

resulting from the contents freezing (dry ice is about 140°F colder than ordinary ice). There is the added danger of contaminating the dry ice and the container. This practice is prohibited and the rule will be enforced.

5 - Use care in handling wrenches. Stools are provided to help reach the upper valves.

6 - Remember that P.G. is present in the manifold and sampling lines and that although it should be duly respected, it should not be feared. White fumes are a sign of danger. Take precautions not to open a valve unless you know which it is and what should happen after the valve is opened. Use care in handling sample tube especially if a leak is suspected.

7 - The Calrod heaters on the "U" tube of the Sampling manifold will give burns, since it heats the tube to a temperature higher than that of boiling water. Turn the heater off when it is not in use.

8 - Handling electrical connections on the tube rack may result in a shock. Aside from changing C.C.R. from one recorder to the other, the operator should have no reason to handle any of the electronic equipment behind the Relay Racks.

These and other safety precautions will be followed by an alert operator. Remember, it's better to be safe than sorry.... [redacted]

2. Equipment & Supplies:

In addition to the relay rack, tube racks, and manifolds, the Line Recorder Stations will be provided with the following equipment:

1 Log table
1 PAX telephone
1 Telephone stand
4 Log Books (2 L.R., 2 Sampling)
4 Data Sheet Clip Boards
2 Chairs
1 Cabinet
1 Foot Stool
1 Tool box with Complete Set of Tools**
2 Sampling Stools
1 Waste can
1 Dry-ice container
1 Scoop
1 Wooden stirring rod
1 Trichlorethylene container (red can)
1 L-28 Transfer apparatus
1 L-28 Carboy (50-liter carboy)
1 L-28 Dispensing can
1 Sampling Equipment (gloves, sample cards, etc.)

Those items which are not present in a station will be supplied as soon as possible. Each operator is expected to keep all equipment in its proper place and in good condition.

The supplies used by each Station will be furnished by a Supply Crew operating from centrally located supply rooms.

** A list of tools will be issued at a later date.

[redacted]

It is the duty of the 8-4 shift to check the inventory of all expendable supplies, such as trichlorethylene, dry ice, L-28, etc. Enough of these materials should be on hand to last until the next week-day 8-4 shift. This is necessary at present since the supply crew works straight day-shift. If urgently necessary, supplies can be obtained on the 4-12 and 12-8 shift by contacting the Section Supervisor who has the supply room key. The supply rooms for the various sections are as follows:

Supply Room	Bell Phone	PAX Phone
301-3	8-9286	45
303-5	8-9411	
305-2		
305-11		

3. Housekeeping

One of the requirements of an A-1 Station is that it be clean and orderly. This includes such items as:

1 - Keeping Log Books and Log Sheets in neat arrangement with no miscellaneous material scattered around.

2 - Disposing of waste such as paper scraps, cigarette butts, lunch bags, etc. into the waste can supplied for this purpose. This also includes magazines and newspapers which are not to be brought into a station.

3 - Placing equipment in the proper location.

[redacted]

This applies particularly to fragile equipment such as Dewar Flasks. Check with supervisor regarding the proper location for the various items of equipment.

4 - Sweeping and general cleaning which will be done by special janitresses who have been assigned for daily clean-up of each Station.

4. <u>Responsibility of Operators</u>

Some general points regarding the responsibilities and duties of L. R. Station operators are:

1 - Operators should make every effort to relieve the previous shift a few minutes ahead of time. At least 10 minutes is desirable since it provides time for the new operator to learn the status of the Station and building before actually starting operation. Thus, the relieved operator is not required to stay late.

2 - If an operator is unable to come to work because of illness or equivalent reason, word should be forwarded to the Main Office (8687) as early as possible so that the necessary relief can be provided. This is also essential if it is necessary to come in late or leave early.

3 - During the last hour of each shift, the operator should determine the status of each cell

[redacted]

in the 2 buildings and the status of each Line Recorder unit in the Station. This information is made available to the supervisor and is also to be entered in the Station Log Books.

4 - Upon coming on shift, the operator should ascertain the building and Station status from the Log Books, Strip Charts, and the previous Station operator and/or supervisor. These communications are an important link between shifts and are essential for smooth round-the-clock operation.

5 - The operator should take readings regularly and promptly (see page 147), and should see that the Station is kept in good order. A good operator keeps <u>well-informed</u> and is <u>constantly alert</u> for any conditions which may require special attention.

6 - The operator is responsible directly to his or her unit supervisor. Any suggestions regarding the operation of that station should be given to this supervisor who will in turn relay them to the operator. This is necessary so that there will be a minimum conflict of authority.

7 - The operator is not obliged to act on orders from building foremen instead, he should refer the foreman to the unit supervisor. In no case should an operator give information (such

[redacted]

as inleakage rates, location of leaks, etc, to building formen or Process technical men. This is the responsibility of the supervisor and of C.C.R. However, the operator can obtain information <u>from</u> the foremen regarding building status, etc. so that this information can be relayed to C.C.R.

8 - The operator should keep the Central Control Room informed of all process changes and any pertinent information concerning the Line Recorders. (See page 22). The C.C.R. can request the operator to make changes in the operation of the "principal recorder" if these are found necessary. However, C.C.R. does not have the authority to do more than advise regarding general Station operation.

9 - In the event of a surge or passage of a slug of contaminant, C.C.R. will notify the operator of its origin and cause as soon as possible. DO NOT CALL OTHER STATIONS because it is imperative that the telephone lines be kept open; instead, contact C.C.R.

10 - The operator should see that proper operating conditions are maintained. This includes seeing that cold traps are filled, that the proper valves are opened or closed (these should be checked at the start of each shift) that the routine checks (discussed earlier in

[redacted]

this section) are properly made.

5. Maintenance and Repair

1 - Preventive Maintenance:

A routine check of various components of the Line Recorder units will often indicate a defective part which may later give trouble. The Instrument Department will therefore have a schedule whereby maintenance men will check every machine daily to see if alarm systems, pumps, electronic units, etc. are properly operating.

These checks are designed so that they will not disrupt normal operation of these units. The instrument is still under control of the operator who can request that no checks interferring with normal operation be run at that time.

2 - Experimental Runs

Various experimental runs may be conducted by maintenance men to obtain information on the operation of a certain unit. All experiments must be sanctioned by the L. R. Department head and work should not be started before contacting the Unit Supervisor. In general, experiments will be conducted only under those conditions when normal operation of the station will not be interfered with.

[redacted]

3 - Installation

New units are installed by a crew from the Instrument Department. When the unit is installed and calibrated, the Unit Supervisor should be contacted for checking the work and signing the Calibration Chart. If the unit is in a building not yet in operation, the Maintenance section should be contacted so that the unit can be kept conditioned with C-616 until such time that the Station is taken over by the L. R. Department.

4 - General Repair

Repair work is often required in the Stations since the equipment used is very delicate and the results must conform to high standards of accuracy. To obtain prompt and efficient repair service, the procedures outlined below should be followed:

1- Notify supervisor
2- The supervisor will help analyze the difficulty
3- A work order is made out and the Maintenance supervisor is contacted
4- Maintenance men take over the machine and proceed with repair work
5- Upon completion, the supervisor checks the work and if found satisfactory, the unit is returned to normal operation.

For a more detailed outline of this procedure, see page 715.

[redacted]DENTIAL

. Shift Schedules and Job Assignment

The assignment of jobs and shifts is made periodically to meet the demands of the work and the number of employees available. Following the training period, each employee is given an opportunity to choose a definite shift and every effort will be made to provide this if possible. However, the supervisor in charge of arranging work schedules reserves the right to make any changes necessary for the benefit of the Department as a whole.

A shift schedule is provided on the following page. Mr. Fleshman or the supervisors will answer any questions regarding the schedule.

7. Intra-Department Coordination

In order to facilitate operations in the Line Recorder stations and to coordinate the work of the L. R. Department on an overall basis, it is necessary to standardize procedures, check the station equipment and collect all pertinent data regarding abnormal operating conditions.

The standardizing of procedures is partially accomplished with the issuing of this manual (providing the operators will follow the instructions given herewith) and as the supervisory and operating personnel become more familiar with the equipment and its eccentricities, new and/or

SHIFT SCHEDULE

Week Starts Monday 12:01 AM
First Shift is Midnight
Second Shift is Day
Third Shift is Evening

During Seven Weeks Cycle

2 Change from Midnights to Evenings	1-80 hrs. off	1-56 hrs. off.
2 Changes from Evenings to Days	2-32 hrs. each	
2 Changes from Days to Midnights	2/32 hrs. each	

Weeks	Job	1 st.Week							2nd. Week							3rd. Week						
Days		M	T	W	T	F	S	S	M	T	W	T	F	S	S	M	T	W	T	F	S	S
12-8	1	R	R	R	C	C	C	C	C	C	C	B	B	B	B	B	B	B	A	A	A	A
Shift	2	D	D	D	R	R	R	F	F	F	F	F	F	F	E	E	E	E	E	E	E	D
8-4	1	C	C	B	B	B	B	B	B	B	A	A	A	A	A	A	A	R	R	R	C	C
Shift	2	F	F	F	F	F	E	E	E	E	E	E	E	D	D	D	D	D	D	D	R	R
4-12	1	B	A	A	A	A	A	A	A	R	R	R	C	C	C	C	C	C	C	B	B	B
Shift	2	E	E	E	E	D	D	D	D	D	D	D	R	R	R	R	F	F	F	F	F	F
Days Off		A	B	C	D	E	F	R	R	A	B	C	D	E	F	F	R	A	B	C	D	E
Jan. 1945																						
Jan. Feb. March		29	30	31	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
March, Apr. May		19	20	21	22	23	24	25	26	27	28	29	30	31	1	2	3	4	5	6	7	8
May, June		7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27
June, July August		25	26	27	28	29	30	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
August Sept.		13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	1	2
Oct. Nov.		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21
Nov. Dec. Jan. '46		19	20	21	22	23	24	25	26	27	28	29	30	1	2	3	4	5	6	7	8	9

Weeks	Job	4th. Week							5th. Week							6th. Week							7th. Week						
Days		M	T	W	T	F	S	S	M	T	W	T	F	S	S	M	T	W	T	F	S	S	M	T	W	T	F	S	S
12-8	1	A	A	A	R	R	R	C	C	C	C	C	C	C	B	B	B	B	B	B	B	A	A	A	A	A	A	A	R
Shift	2	D	D	D	D	D	D	R	R	R	R	F	F	F	F	F	F	F	E	E	E	E	E	E	E	D	D	D	D
8-4	1	C	C	C	C	C	B	B	B	B	B	B	B	A	A	A	A	A	A	A	R	R	R	R	C	C	C	C	C
Shift	2	R	R	F	F	F	F	F	F	F	E	E	E	E	E	E	E	D	D	D	D	D	D	D	R	R	R	F	F
4-12	1	B	B	B	B	A	A	A	A	A	A	A	R	R	R	R	C	C	C	C	C	C	C	B	B	B	B	B	B
Shift	2	F	E	E	E	E	E	E	E	D	D	D	D	D	D	D	R	R	R	F	F	F	F	F	F	F	E	E	E
Days Off		E	F	R	A	B	C	D	D	E	F	R	A	B	C	C	D	E	F	R	A	B	B	C	D	E	F	R	A
Jan. 1945		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
Jan. Feb. March		19	20	21	22	23	24	25	26	27	28		2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
March, Apr. May		9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	1	2	3	4	5	6
May, June		28	29	30	31	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
June, July August		16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	1	2	3	4	5	6	7	8	9	10	11	12
August Sept.		3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
Oct. Nov.		22	23	24	25	26	27	28	29	30	31	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Nov. Dec. Jan. '46		10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	1	2	3	4	5	6

revised procedures will be issued.

Checking the status of buildings and of station equipment will be done once per day by the Intra-Department Coordinator. However, for a continual check, it is necessary that the operators call the Records Department of the C.C.R. and notify them of the following conditions:

1 - Cells off stream

a// For tests
b// For repairs

2 - Stations with faulty equipment

a// Tube racks
b// Relay racks
c// Manifolds
d// Sampling Manifolds

3 - Any time when the equipment listed above is repaired or put back on-stream.

This data is to facilitate the repairing of equipment and for maintaining a better over-all picture of the Cascade.

8. <u>Questions and Answers</u>

Some of the many questions asked of Mr. Fleishman by operators are included below:

Q. What must I do if I fail to clock in or out?

A. On the following day, a notice will be attached to your clock card indicating failure to clock in or out. Remove this notice and write on the back thereof the reason for the failure and the actual time of leaving or arriving. Turn this

 [redacted]

card over to Mr. Fleshman. Unless this is done, loss of a day's pay will result.

Q. How may a discrepancy in my pay check be corrected?

A. Bring the check or stub to Mr. Fleshman and explain what's wrong. He either will get it adjusted or send you to the proper place to get this done. Or he may offer a satisfactory explanation regarding an apparent discrepancy.

Q. What should I do if I expect to be absent?

A. Call 8687 as directed on page______. An absentee Log Book will be provided near that phone. Any absence not logged in that book will be forwarded in as an unexcused absence.

Q. How do I get my shift or job assignment changed?

A. Make out a written request and submit this to Mr. Fleshman. List all reasons for the request. All possible consideration will be given.

Q. What if I have a complaint or grievance?

A. See Mr. Fleshman personally and he will see that the matter is taken care of to your satisfaction.

Q. Where do I get information regarding salary and raises in pay?

A. Carbide has a standard system of merit increases in pay rate and Mr. Fleshman will gladly discuss this matter with any of you. In the Line Recorder Department there are two general classifications for operators:

[redacted]

B operator - requires supervision
A operator - requires no supervision and has the ability to teach proper operation to new operators.

The pay rates of these classifications are approximately the same as the A & B classifications throughout the plant.

Q. When do I get a vacation?

A. Carbide policy regarding vacations for hourly employees is that they get 1 week per year at straight-time pay. You are eligible after completing one year of continuous service. See Mr. Fleshman about vacation schedules.

Q. What about overtime work?

A. Overtime work is granted to any employee if the need is justified. No limit on the amount worked by male employees, but women may not work more than 4 hours overtime per day or more than 66 hours per week. Overtime is paid for all work over 8 hours in any constituted working day or over 40 hours in any constituted working week. No overtime is paid unless special authorization is signed by Mr. Fleshman and turned into the time-keeping department.

Other questions on insurance, sick leave, etc. should be taken up with (Dr.) Fleshman.

[redacted]

c. Telephone System

The Line Recorder Stations are provided with an inter-station PAX system. Calls should be of limited length so that the lines are kept clear in case of an emergency. A list of numbers follows:

PAX SYSTEM

311-1 310-3	10
310-1-2	11
309-2-3	12
309-1 301-1	13
301-2-3	14
301-4-5	15
302-1-2	16
302-3-4	17
302-5 303-1	18
303-2-3	19
303-4-5	20
303-6-7	21
303-8-9	22
303-10	40
Central Control Room (General)	23
Central Control Room (Emergency)	24
Central Control Room (Records)	25
304-1-2	57
304-5 305-1	26
305-2-3	27
305-4-5	28
305-6-7	29
305-8-9	30
305-10-11	31
305-12 306-1	32
306-2-3	33
306-4-5	34
306-6-7	35
312-1-2	36
312-3	37
H. A. Nowak - Office	58
Mayer's Office	38
Area Office 302-4-5	43
Tevis, Drukey - Office	39
L. R. Maintenance Shop 301-3	42
Switchboard Operator	44
L. R. Supply Room 301-2	45

Bell Phone System: Main Office 8687
C.C.R. 8-9457

[illegible] between the operators, the supervisors and [illegible] usually be required during non-routine procedures.

Although each situation presents its own individual problems, it is hoped that the following discussions will help prepare both the operator and the supervisor for the proper handling of any such abnormal situation.

B. Process Leaks

To give an idea of the unprecedented tightness which all process equipment in the K-25 plant must reach, the following is quoted from the OPERATIONS MANUAL, 300 Section, Volume II, Page 276: "If the whole plant were completed and every part in it were absolutely tight and a hole were drilled somewhere in one pipe with a No. 80 drill (about 15thousandths of an inch in diameter), the plant would then have the overall inleakage which is the maximum it may have when finished." Only a small number of tiny leaks are needed to exceed the maximum allowable inleakage.

As stated on Page 2, the principal functions of the Line Recorder are to determine whether or not the leakage of G-74, air, or C-816 into the C-616 is normal, and to assist in locating an abnormal leak in the system.

C[redacted]DENTIAL

The operator should therefore be sufficiently acquainted with Process and Line Recorder operation so that a leak can be recognized and then isolated.

a. G-74 and Air Leaks

The normal inleakage of G-74 into the process stream results in a small concentration of this contaminant throughout the plant with little variation from one cell to another. The G-74 passes to the top of the Cascade and is removed from the Cascade by continuous purging. This normal G-74 inleakage results from the following sources:

1 - Seal Feed system
2 - Returning off-stream equipment to process
3 - Single-seated block valves isolating process from G-74 supply
4 - Double-seated block valves pressured with G-74 when equipment is down for repairs
5 - Leaks from cell (ambient) air

G-74 is also used in the datum and buffer zone systems and for purging equipment to remove C-616. With this widespread use of G-74, there are many potential sources of abnormal G-74 inleakage into the Process stream.

Source (5) introduces air into the C-616 so a rise in the 32 signal would distinguish this type of leak. Since the outer seal of the pumps is supplied with dry air, there is also a possibility of dry air entering P.G. lines in case of seal failures. Also cross-overs from the dry air system to the G-74 headers may introduce air into seal feed storage drums if they are

[redacted]

opened accidentally or intentionally.

A leak in the seal feed system may be due to a high seal feed pressure which would be indicated on the cell panel board. If the seal exhaust pressure is high, more G-74 is admitted into the process stream and this will account for a G-74 rise.

When offstream equipment is returned to the cascade, a packet of G-74 is usually introduced. Often a G-74 packet is deliberately introduced into the cascade as when the contents of a cell or surge drum are emptied into the stream. In these cases, either the Station operator or C.C.R. should be notified beforehand by the men in charge of that operation.

Single and double-seated block valves are a common source of G-74 inleakage. In case of a G-74 leak through a valve bonnet, it is quite possible that the leak is stopped when the block valve is closed and started again when the valve is opened. This will mean that the leak may "disappear" as soon as the cell is turned on inverse recycle.

Leaks from the ambient air are evidenced by a rise in the 32 mass signal as well as a G-74 rise. If a "wet air" leak occurs, the 20 mass signal may also rise although this has not yet proved too reliable.

Since G-74 is used in the datum and buffer zone systems, these represent another possible source of inleakage (through the Stage pressure tap).

[redacted]

These will be evidenced by a drop in datum pressure or a rise in the buffer zone flow. The use of G-74 as a purge gas provides many sources of possible in-leakage since the purge lines are separated from onstream equipment only by single-seated block valves. Offstream equipment being pressured with G-74 may not be tightly isolated from onstream equipment so it is well to be informed whenever G-74 is admitted into any process piping, whether on or offstream.

Since both G-74 and oxygen are much lighter than C-616, packets tend to travel up the cascade fairly rapidly. Present information indicates that the rate of travel of G-74 or air slugs is about 2 cells per minute (26-28 seconds per cell).

With the aid of the cell panel boards, and information from C.C.R. or men in charge of building operation, the experienced operator can often determine the source and magnitude of G-74 or air leaks. The presence of a leak is unlikely if the G-74 reading returns to its former value after a few minutes. The next higher onstream recorder should also indicate a G-74 rise if a leak actually is present. The leaks are located by scanning as will be described below.

If an increase of G-74 or G-74 and oxygen is noted, check to see if the rise is indicated on the next higher Line Recorder. If not, a leaking manifold (this may cause a <u>slight</u> rise in the next recorder reading)

[redacted]

or a leaking tube rack may be the cause. A rise in the M.S. tube pressure (check ion gage reading) or a defective electronic unit may also cause high readings. If the amplifier zero or the Pirani reading is high, an apparent increase in the G-74 or oxygen concentrations will result. If in doubt regarding the cause of the rise, contact the supervisor. It is often possible to obtain useful information by checking the "principal recorder" against the "spare".

A change in process operation may be the cause of a G-74 rise in that cell. If the cell pressure is reduced or the Direct Recycle valve is opened, the G-74 concentration increases. This condition may be prevalent between two sections operating at different conditions. The cell panel board should be checked in these cases. The operator should be well-informed on the various types of process operation, and in particular on the type of operation being used in the buildings analyzed by that Station.

If a G-74 or air leak is suspected, contact the supervisor and C.C.R. at once. Determine the per cent rise in the G-74 concentration from the 14 and the 28 signals. This may be done as follows:

Former Reading (14 peak)	3 divisions x 1 x .02 (const.)
New Reading	8 divisions x 1 x .02

Per cent rise in G-74 concentration is .06% to .16% or <u>.10% rise</u>.

The "critical" rise in concentration differs from one component to another and from one set of operating conditions to another, so a system of letters is used in the

[redacted]

following discussion rather than actual figures. The table included below should be filled in (with pencil) with the proper critical values which will be issued as soon as available:

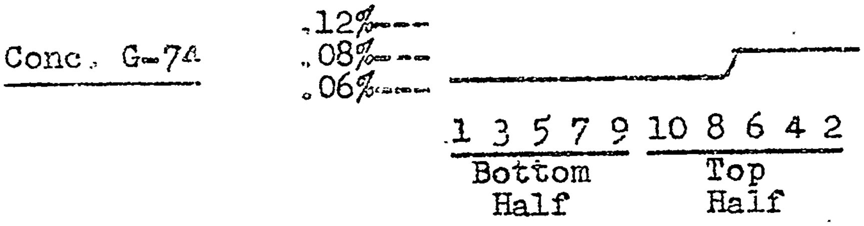
SECTION	G-74		C-816	
	X%	Y%	L%	H%
311				
310				
309				
301				
302				
303				
304				
305				
306				
Purge				

For air or G-74 leaks, rises in the G-74 concentration below X% indicates a small leak. Scanning should be promptly begun (see table on page 7//a) but care should be exercised since accurate results are necessary to detect small leaks. Between X and Y% increase, the leak is fairly large and scanning should be started at once. Above Y% increase, the leak is sufficiently large to warrant placing the building on Inverse Recycle. If this does not cause the G-74 concentration in higher buildings to drop, the next higher building and, if necessary, the next lower building should be put on Inverse Recycle. Check with C.C.R. in all cases.

If it is not certain that the leak is in a certain building, check the top cell against the bottom cell. If a

[redacted]

significant difference is observed, scan as indicated on the chart. The following is included as an illustration:



(10-cell building; no Inter-sectional cells)

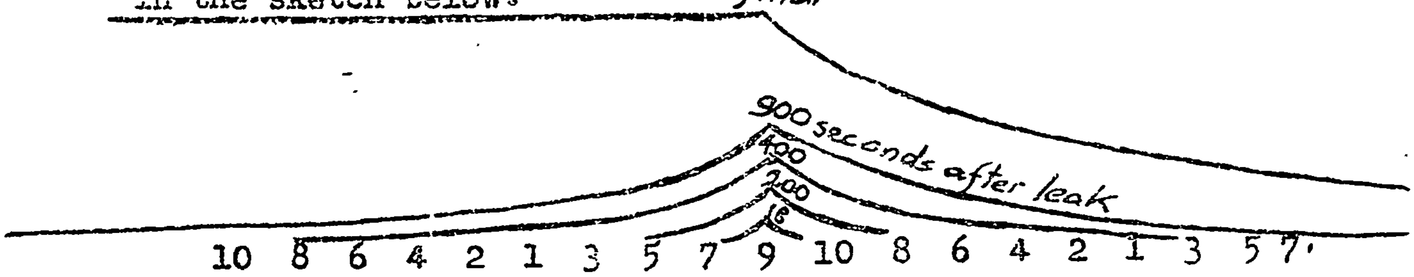
In this case, a top & bottom scan would result in a high reading from cell 2 and a low reading from cell 1; cell 10 would give a low reading so the leak is isolated to the top half; cell 6 reads high and cell 8 reads low, so the leak may be assumed to be in cell 6. However, the leak may also be in the by-pass lines or in cell 4. This can be determined by checking cell 4 and/or putting cell 6 on Inverse Recycle. If the by-pass lines leak, the building should be put on Inverse Recycle to keep G-74 from contaminating the rest of the cascade. If careful checking has been followed and conditions seem to justify putting a cell or building on Inverse Recycle, the building foreman should be notified to take the proper action. This should be done <u>only</u> by the supervisor or by C.C.R.

b. <u>C-816 Leaks</u>

Since C-816 is slightly heavier than C-616, its behavior in the process stream is considerably different from that of the light contaminants. It travels very slowly spreading in both directions with slightly more travelling towards the bottom of the cascade. At the start, one cell will have a high concentration of 816 and its presence can be determined by scanning the building.

 [redacted]

Later, if the leak is allowed to continue, the cells both above and below the leak will have high 816 concentrations with a near-horizontal plateau in the lower cells as shown in the sketch below:



The C-816 spreads out in a manner analogous to that of sand being poured slowly on a sand pile. The exact location of the leak is more easily determined at the early stages than later when many more cells have been affected.

If an 816 leak is suspected, it is first advisable to check the instrument before assuming that the 816 is actually present in the cell. If the L-28 cold trap on the tube rack is not operating properly, condensed 816 may diffuse back into the M.S. tube and cause a rise in the 69 signal. If mercury diffuses into the M.S. tube from the chemical trap or from the diffusion pump via the L-28 trap, the 69 peak may appear to rise (especially if the instrument is slightly off peak, since the 67 peak actually rises).

If an 816 leak is actually present, it should show up on more than one recorder. Place the spare recorder on the same cell if necessary to check the other unit. The leak is small if the rise in 816 concentration is below 1% (see p. 206). If the leak is detected at an early stage, scan to locate the

[redacted]

cell with the highest 816 concentration (taking [illegible] the change of peak with time as shown on previous page.) If the leak is noted after an hour or more, scan cells to determine the top cell of the plateau.

If the rise in 816 concentration is between L and H%, scan cells at once as described previously. Have the foreman check the coolant levels while scanning. The levels may not have dropped appreciably or a drop may be due to temperature changes, so this test is not too reliable. For large leaks, however, this may often confirm the results of scanning. If the rise is above H%, contact the building foreman at once to have the suspected cells or building(s) taken offstream. It may take some time for the 816 in the onsteam equipment to travel towards the bottom and confirm that the leak has been isolated to a cell or building put on Inverse Recycle. However, these concentrations will drop slowly if the leak actually has been isolated.

c. <u>Miscellaneous Leaks</u>

In addition to G-74, air, and C-816, contaminants which may be detected with the Line Recorder include several other "lights".

1- A rise in the 44 peak may be due to either a carbon dioxide or a nitrous oxide leak from the refrigeration systems. To distinguish between these, the supervisor should check the 30 peak (NO) using the manual divider. Carbon dioxide is being considered as a possible probe gas for spotting leaks. More specific information on this subject will be issued later.

2- A rise in the 20 peak may be due to a "wet air" leak or a drop in the feed purity. This rise may be caused by insufficient S-44 in the slush trap.

[redacted]

3- A rise in the 28 peak is usually accompanied by a corresponding rise in the 14 peak. However, the 28 peak may rise from CO or from N_2O, although this is uncommon. The 28 peak is much more sensitive than the 14 peak and small leaks can often be detected in this manner when no rise in the 14 signal is noticeable.

The program of the instrument can be changed so that other suspected impurities can be analyzed if deemed necessary.

C. Manifold and Tube Rack Leaks

A suspected process leak often turns out to be a leak between process and the M.S. tube, either in the manifold or the tube rack. The leak is usually located without difficulty if a logical procedure is followed. The manifold diagram is a valuable aid in determining the exact source of inleakage. Some common sources of inleakage follow:

1- Sample lines from the 6A discharge to the manifold and back to the 6A suction may leak. This is evidenced by the fact that the analysis from one cell seems to be "out-of-line" with the others. The G-74 and oxygen concentrations appear higher with the leaking cell than with the others. These leaks may cause a slight rise in the next higher cell since the contaminants are being returned to the process stream.

2-Manifold loops above the adjustable leak may have a leak which is indicated by a change from normal analysis when the crossovers are used. The same L.R. unit will have different

[redacted]

readings if switched to the non-leaking loop. Since there are no crossovers on the line below the adjustable leak, it is necessary to close all valves and evacuate the loop (using valve 62 or 63) and proceed to locate the leak by isolating different sections of the evacuated line. This procedure is followed when the manifold loops are suspected of leaking and no change is observed upon switching to the other loop.

3- Leaks behind the adjustable calibration leak are common. Even if this leak is closed tightly and two block valves are also (supposedly) tight, inleakage into the sample stream is often possible. Pumping down behind the leak (see page 114) should bring the readings down.

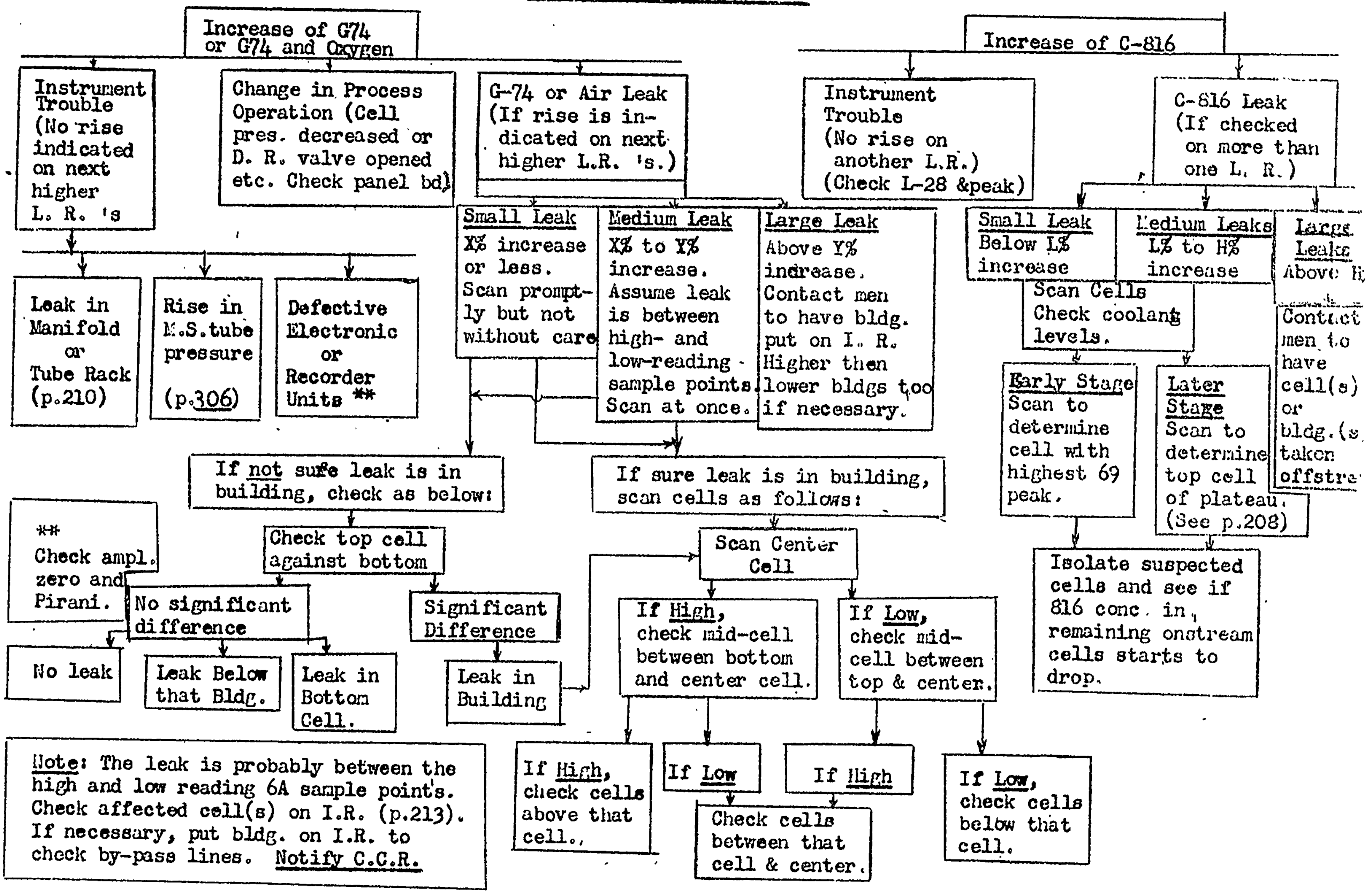
4- Seat leakage of various valves is common. This may be spotted when different readings are obtained using lines with non-leaking valves.

5- Sampling manifold leaks occur quite often. These are evidenced by the difficulty in getting this system pumped down and the quick rise in pressure after the evacuated system is isolated from the pump. The gas passing to the tube rack is often contaminated by leakage through the T-valves 68 or 69.

6- Tube rack leaks are evidenced by high G-74 or oxygen readings and by high residual values. It is difficult in this case to get very low pressures in the M.S. tube. The pressure may rise to the point where the filaments are tripped off. Similar results will be obtained if the diffusion pump, mechanical pump, or cold traps are not operating properly.

[redacted]

LEAK DETECTION PROCEDURES



[redacted]

There are many more possible sources of inleakage on the manifold and tube rack, but these can best be located by examining the facts available and by conducting some simple tests. If both tube racks indicate a leak which is not a process leak, the following systems should be checked since each of these is connected to both analyzing systems:

a- Sampling Manifold
b- Manifold Evacuation Line
c- Calibration Lines

These systems are checked by evacuating and isolating various sections of the suspected system until the leak is located. When the leak is located (or if the exact location cannot be determined, let Maintenance men find it), the supervisor should make out a work order to have the defective part repaired or replaced. (See page 215).

D. Inverse Recycle Operation

1. Routine Checks

In order to distribute the program of cell repair evenly over a period of time, routine checks of the inleakage rates of G-74, air, and C-816 will be conducted on cells operating on Inverse Recycle. A group of the L. R. department is being set up to collect this information and to forward it to the V. T. D. which functions to keep plant equipment tight. These routine checks will be supervised by the aforementioned group. Additional information from cells operating on Inverse Recycle for other routine checks or repairs should be obtained whenever possible and the results will be collected by the above group.

[redacted]

2. Confirming Suspected Leaks

In order to confirm or refute the suspicion that a certain cell is leaking, it should be placed on Inverse Recycle to determine whether the inleakage is normal or above normal. Sinee the cell is a closed system with no continuous G-74 purge, the inleakage will cause a gradual rise in the G-74 concentration as well as the average pressure of the cell. If the oxygen shows a gradual rise also, an air leak may be suspected.

The per cent increase per hour of G-74 concentration should be calculated after the Line Recorder has been analyzing the cell for at least 30 minutes. Readings should be corrected for amplifier zero and Pirani deviations. Use the following equations:

Corrected Signal = (Reading x Sens. Factor) - Ampl. Zero

Concentration = Corrected Signal x Constant

$$\% \text{ Increase/Hr.} = \frac{\text{Conc.}_2 - \text{Conc.}_1}{\text{Time Interval (Hours)}}$$

The constant is obtained from the Calibration chart posted above that L. R. Unit. If the Pirani reading is different from the proper value for that concentration (see page 105), correct the signal by multiplying it by the ratio of correct Pirani value/actual Pirani reading. Although the above calculations may seem somewhat complex, they should be used if accuracy is required. Be sure that the machine is peaked and recently calibrated or the readings may be off.

The actual inleakage rate can be calculated with fair accuracy (within 10 per cent) with the following formula:

Inl. Rate = % increase per hour x F x Cell pressure

[redacted]

The cell pressure is the average of the high-side pressures of all the stages of that cell over the period that the change in concentration was determined The value of F for different sections is given in the table below. Also given are values for low inleakage and maximum allowable inleakage for cells in good condition. Consult with C.C.R. before deciding that the inleakage is sufficiently great to warrant shutting the cell down for repairs. The value of F is different at higher G-74 concentrations. It is advisable to take all inleakage rates at low G-74 concentrations since there is a greater change in signal per unit inleakage at the lower concentrations. Results at high G-74 concentrations are often unreliable.

SECTION	F	Low Inleakage	Max. Inl.
311			
310			
309			
301			
302			
303			
304			
305			
306			
Purge			

[redacted]

I. Maintenance Repair.

a. Procedure for Ordinary Repair Work.

Many troubles arise in the Line Recorder stations which require the assistance of the repair crews from the Maintenance Department. A few of these troubles are:

1. Leaky Manifold lines or valves.
2. Defective valves (Crane "T", etc.).
3. Sampling Manifold leaks.
4. Cracked M.S. tube (glassware).
5. Scatter of print points.
6. Defective or incorrect Pirani readings.
7. Amplifier out of adjustment.
8. Loss of vacuum with the M.S. tube.
9. Contaminated calibration samples.
10. Micromax not printing correct readings as compared to the Amplifier Zero or Pirani gage.

Or any trouble that will cause error in data taken.

To obtain prompt and efficient repair service it will be the responsibility of the operator to notify the shift supervisor of the trouble. The shift supervisor will diagnose the trouble to the best of his ability and if assistance is required will contact the Section Supervisor.

Upon determining the necessary repair a work supplement order shall be made out by the supervisor

CONF[redacted]

or man in charge of work orders to the maintenance repair crew.

The correct procedure on writing a Repair Order Supplement Form (WCX - 170) should be as follows:

1. Fill out the order in triplicate.
2. File the blue copy.
3. Give the white and pink copies to the Instrument Dept. Maintenance Shift Supervisor. (It has been decided that the L.R. supervisor will either leave the Repair Order Supplement Form in the station that needs repair and then notify the I.D. supervisor or take the forms to the I.D. Supervisors' Office.*
4. When the maintenance man arrives to do the necessary work the L. R. operator must receive the supervisor's permission to allow the men to work.
5. The operator will enter the form number and the time that work started in the Station Log Book.
6. A "Maintenance" tag is put in the window of the Micromax of the troubled machine. (This is at times not necessary if the machine can be kept operating.)
7. When the work is completed the operator shall notify the Section Supervisor. He will check the repair job and if satisfactory sign the white copy of the order. This then certifies that the work was properly done and checked.

8. The operator will enter the time that the job was completed and the Repair Order number in the station Log Book.

At the end of the day, all pink slips will either be mailed to us or the Section Supervisor can collect them at the I.D. Supervisors' office.

B. Acceptance of a Recalibrated Machine.

When a Line Recorder has been calibrated, the Calibrating Maintenance Crew will contact the shift supervisor for that particular building and turn the machine over to him.

The shift supervisor will then run a 95-5 Check (See Page 117) and if the machine proves satisfactory, it will be accepted. Acceptance of a machine is denoted by the signing of the Calibration Constant Chart.

[redacted]

Acceptance Of A Machine With A Replaced Panel

Whenever a Line Recorder has a faulty panel the panel will be replaced with one that has been checked and approved. However, this new panel may differ in regards to the old panel enough to effect the calibration of the machine

To check the calibration it is suggested that the machine be left running on the same cell during replacement of the panel, and then noting if the readings for the different masses correspond closely before and after the machine was repaired. If the readings do not coincide, a 95-5 or *95-3-2 check should be run to determine exactly the accuracy of the machine.

* A 95-3-2 check is run the same as the 95-5 check. (It contains 95% C-616, 3% G-74, & 2% C-816.)

[redacted]

EMERGENCY PROCEDURES

A. Power Failures

When the electric power to the L. R. equipment fails, the operator will know immediately since all of the pilot lights will go out, the output meter on the D. C. Amplifier Panel (if on) will read off scale, all the fore pumps will stop and in some cases the ceiling lights will go out.

In the case of a power failure, the precautions which are to be taken are fairly simple. The dangers that may be encountered are the loss of vacuum with oil being drawn into the fore pump, and damage to the amplifier by the draining of the output batteries or burning-up of the output meter.

To prevent this damage:

1 - Close the shut-off valves to the fore pumps of each tube rack.

2 - Open the fore pump vent valves on each tube rack momentarily and then close them.

3 - Close valve 152 on each tube rack.

4 - Close valves 95 and 96 of the manifold pumps.

5 - Open valve 94.

6 - Turn output meter sensitivity switch to "off".

7 - Turn the channel sensitivity switches to "off".

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8 - Turn the Acceleration Voltage Selector switch to the "off" position.

9 - Turn the M. S. tube filament switch to "off".

When the Power returns and the pilot lights are on, the procedures are:

1 - Check all vent valves on Tube Racks and manifolds.

2 - Start up fore pumps.

3 - When pressure at the fore pump suction (See T.C. #2) is less than 15 microns and the mercury has been distilling over for 5 minutes or more, open the bypass valve on the tube rack. Close the shut-off valve between the diffusion pump and the mechanical pump.

4 - Pump the line behind the adjustable leak down to 15 microns. Close the by-pass valve, and open the fore pump shut-off valves. This should be done only if the fore pump pressure (T.C. #2) is approximately the same or lower than the diffusion pump pressure (T.C. #1).

5 - Check the M.S. tube pressure by means of the Ion Gage. If this pressure is 1×10^{-6} mm of Hg. or less, open valve 152 slowly so that the Ion Gage does not exceed 1×10^{-5}.

6 - Open valves 152, 95 and 96.

7 - Turn the Acceleration Voltage Selector switch to "Record".

[redacted]

8 - Switch on the M. S. tube filament.

9 - Adjust the sensitivity controls to their proper value.

B. <u>Water Supply Failures</u>

The water supply for the L. R. equipment is provided by the sanitary water system which is at times subject to failure. When this happens there is danger in loss of vacuum, loss of an excessive amount of mercury and in possible cracking of the water jacket that surrounds the diffusion pump.

In such cases, the action taken for the safe-guarding of station equipment will be:

1 - When the operator notes that the water supply has failed, he will immediately notify his shift supervisor.

2 - Upon determination that the water supply will be off for more than 5 minutes the operator will:

a. Turn off the heater current to all diffusion pumps (tube racks and manifold).

b. Isolate all tube rack pump set-ups in the station by closing valve 152, and 96 for the manifold pump.

c. Cut the M. S. tube and Ion Gage filament supplies.

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d. Check all cold traps and slush traps for proper coolant level - if quite low, refill or call the cold trap operator.

E. After the Diffusion Pump heaters have been off for a period of 1/2 hour or more, close the fore pump, shut off valves.

When the water supply is replenished or repaired:

3. Switch on the heater currents and allow the diffusion pumps to heat until the mercury has distilled over for at least 5 minutes, then open the by-pass valve on the tube rack. Pump the line behind the adjustable leak down to 15 microns, close the by-pass valve and open the fore pump shut-off valves. This should be done only if the fore pump (T.C. #2) is approximately the same or lower than the diffusion pump pressure (T. C. #1).

4. When these conditions have been obtained open the fore pump shut-off valve for the manifold pumps as described above and valve 96.

5. Check the M. S. tube pressure by means of Ion Gauge. If this pressure is 1×10^{-6} mm of Hg. or less, open valve 152 slowly so that Ion Gage does not exceed 5×10^{-5} mm of Hg.

6. After the pressure in the M. S. tube has decreased to the value it originally was at before the water failure, switch the filaments on and adjust sensitivities to meet the new readings.

If for any reason the tube gives erroneous readings as compared to the previous ones (after a period of 1 hour), maintenance should be contacted through the shift supervisor.

[redacted]

[illegible]

In [illegible] injury or ill[illegible] [illegible] operator during [illegible] [illegible] be taken:

1 - [illegible]

[illegible] will then -

(a) Obtain transportation to the dispensary.

(b) Fill out a dispensary pass.

(c) Get all information necessary for an illness or accident report. (Company policy states that a complete report must be turned in within 24 hours). This information is forwarded to Mr. Fleshman.

(d) Provide a relief operator.

2 - Keep calm as in any emergency. Remember that most supervisors are experienced in First Aid measures.

3 - The Ladies' change houses are provided with cots and attendants for women too ill to continue working. Don't hesitate to notify your supervisor who will see that relief is made available.

4 - In the case the injury is of a minor nature, the particular job (such as sampling, closing a valve, etc.) should be completed. Always notify your supervisor or relief operator of the Station Status so that the work may be continued properly.

CONFIDENTIAL

D Loss of Vacuum due to Tube Fractures.

As with all equipment, accidental damage will occur even when the operating personnel may be as careful as it is humanly possible to be.

In a case where the M.S. tube has been fractured and the pressure has risen so that the filaments cut-off, the following procedure is used:

1. Turn the High Voltage supply and Emission Regulator to <u>Off</u>.
2. Close valve 152 on the Tube Rack.
3. Close down the adjustable leak.
4. Close the fore pump line valve.
5. Bleed the fore pump to atmosphere.
6. Turn the heater current of the Diffusion Pump off.
7. Notify your supervisor so he may call maintenance.
8. Tag the machine "Maint."

Some of the symptoms that indicate a high tube pressure are:

1. Filaments cutting off.
2. Arcing of the High Voltage across the Acceleration plates.
3. Bubbling noise from the fore-pump.
4. In extreme cases, the hissing of air that is leaking into the tube.

[redacted]

B Failure of an Electronic Unit

Of the five main electronic units the D.C. Amplifier & Micromax Recorder are the two that require the most repair work. In case these or the other units become defective the procedure will be as follows:

1. Notify your supervisor immediately.
2. Check thoroughly all units as described previously and then shut off the High Voltage supply and the Emission Regulator Filament.
3. Turn all sensitivities of the recorder to Off.
4. Keep a careful check on the M.S. tube pressure by reading the Ion gage quite frequently.
5. Allow maintenance to check the machine as soon as they arrive. (This duty should be taken care of by the supervisor.)

Symptoms that indicate a defective electronic circuit are numerous, but a few are:

1. Extremely Low or High recorder readings.
2. Scatter of print points.
3. Fluctuating Emission reading.

[redacted]

INDEX